Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Organocatalytic and enantioselective [4 + 2] cyclization between hydroxymaleimides and ortho-Hydroxyphenyl para-quinone methides-selective preparation of chiral hemiketals

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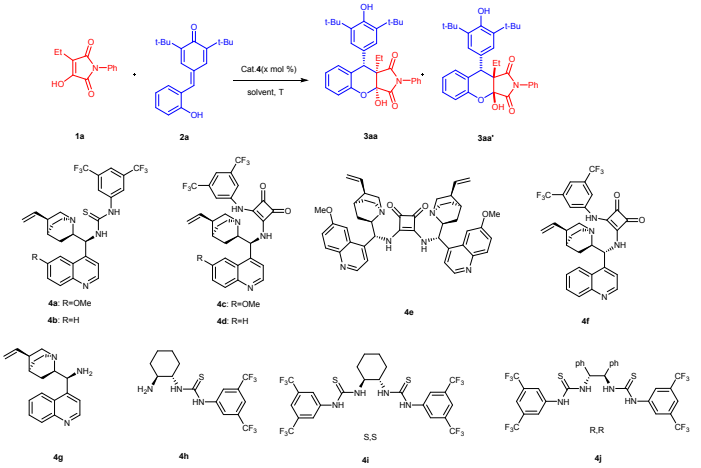
1 General remarks

Chemicals were purchased from commercial suppliers and used without further purification unless otherwise stated. Reactions were monitored by TLC and visualized with ultraviolet light. Flash column chromatography was performed on silica gels (300-400 mesh) eluting with ethyl acetate and petroleum ether. ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ or DMSO-d_6 on a Bruker Avance instrument (400 MHz for ^{1}H NMR,100 MHz for ^{13}C NMR; 300 MHz for ^{1}H NMR, 75 MHz for ^{13}C NMR). ^{1}H NMR chemical shifts are reported in ppm relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard ($CDCl_3$ at 7.26 ppm, DMSO-d_6 at 2.50 ppm), chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ^{13}C NMR chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard ($CDCl_3$ at 77.16 ppm, DMSO-d_6 at 39.52 ppm). High-resolution mass spectra (HRMS) analyses were obtained with the Bruker SolariX 70 Fourier-transform mass spectrometer. Enantiomeric excess was determined by HPLC analysis on chiralpak AD-H and AS-H etc. Optical rotations were measured on a Perkin-Elmer 241 Polarimeter. Melting points were recorded on a Buchi Melting Point B-545.

2 Optimizations of the reaction condition

Table S1. Optimization of the reaction conditions.[a]



Entry	Cat. / x	solvent	Temp (°C)	Time(h	Yield	%[b]	dr[c]	ee %[d]
1	-	DCM	25	1	99		90:10	0
2	**4a**/10	DCM	25	0.1	99		76:24	65/13
3	**4b**/10	DCM	25	0.1	99		79:21	66/0
4	**4c**/10	DCM	25	0.1	99		73:27	85/11
5	**4d**/10	DCM	25	0.1	99		73:27	87/0
6	**4e**/10	DCM	25	0.5	88		81:19	67/28
7	**4f**/10	DCM	25	0.1	99		78:22	-75/19

Entry	Cat. / x	solvent	Temp (°C)	Time(h	Yield %[b]	dr[c]	ee %[d]
8	**4g**/10	DCM	25	3	90	76:24	10/0
9	**4h**/10	DCM	25	0.5	97	67:33	19/0
10	**4j**/10	DCM	25	0.5	97	80:20	-7//0
11	**4d**/10	$CHCl_3$	25	0.25	99	78:22	76/-
12	**4d**/10	DCE	25	1.5	93	70:30	38/-
13	**4d**/10	MTBE	25	4	95	85:15	98/-
14	**4d**/10	THF	25	9	76	84:16	98/-
15	**4d**/10	Et_2O	25	4	99	86:14	98/-
16	**4d**/10	1,4-Dioxane	25	4	99	87:13	98/-
17	**4d**/10	Toluene	25	0.5	99	74:26	69/-
18	**4d**/10	n-hexane	25	168	NR	-	-
19	**4d**/10	MeCN	25	12	98	73:27	75/-
20	**4d**/10	EtOH	25	4	99	66:34	36/-
21	**4d**/5	1,4-Dioxane	25	14	99	88:12	>99/-
22	**4d**/1	1,4-Dioxane	25	60	90	79:21	94/-
23	**4d**/5	MTBE	0	96	90	85:15	91/-
24	**4d**/5	1,4-Dioxane	50	9	98	81:19	98/-
25	**4d**/5	1,4-Dioxane	100	6	95	79:21	87/-
26	**4f**/5	1,4-Dioxane	25	15	94	80:20	-98/-
27	**-**	1,4-Dioxane	25	120	-	-	-

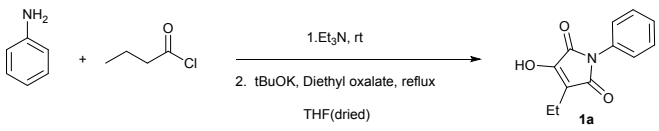
[a]Reaction conditions: **1a** (0.1 mmol), **2a** (0.12 mmol), and catalyst **4** (x mol%) in solvent (0.1 M), at 25 °C. [b]isolated yield. [c]Determined by HPLC. [d]Determined by chiral HPLC, Ar=3,5-di-tert-butyl-4-hydroxyphenyl.

3 General procedure for the syntheses of compounds **1a-1u** and **2a**-**2i**, **5a**,**5b**,**5c**, and catalysts **4**.

3.1 Hydroxymaleimides:1a-1u and characterization data

1a-1u were prepared according to the literature.[1]

Typical preparation for 3-ethyl-4-hydroxy-1-phenyl-1H-pyrrole-2,5-dione **1a.**

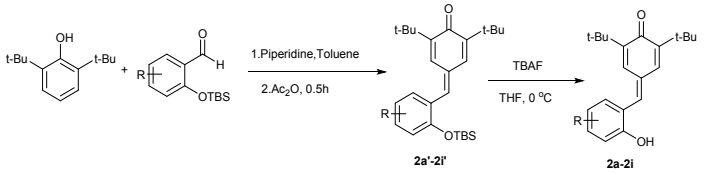


Step-1: A solution of aniline (1.06 g, 10 mmol, 1 equiv.) and triethylamine (1.21 g, 12 mmol, 1.2 equiv.) in 30 mL THF was placed in a round-bottomed flask which was stirred at 25 °C for 10 min, N-butyryl

chloride (1.16 g, 11 mmol, 1.1 equiv.) is injected into the sealed bottle by a syringe and stirred at 25 °C for 3 h.

Step-2: Potassium tert-butoxide (2.80 g, 25 mmol, 2.5 equiv.) was slowly added to the reaction flask and stirred at 25 °C for 10 minutes, diethyl oxalate was added (3.65 g, 25 mmol, 2.5 equiv.) and stirred for 10 minutes. The reaction temperature was raised to 70 °C and stirred for another 12 h. After full consumption of the intermediate monitored by TLC. 2 M HCl (25 mL) was added to neutralize the residue to pH = 1. The aqueous solution was extracted with CH_2Cl_2 (3×50 mL). The organic phases were combined, washed with brine and dried over anhydrous Na_2SO_4. After evaporation of solvent under reduced pressure, the crude product was purified by flash column chromatography (petroleum ether/ethyl acetate = 15/1 to 4/1) to afford product **1a** (Yellow solid, 1.35g, 67% yield). **^{1}H NMR** (300 MHz, DMSO-d6) δ 13.63 (s, 1H), 7.49-7.40 (m, 2H), 7.37-7.28 (m, 3H), 2.30 (q, J = 7.5 Hz, 2H), 1.08 (t, J = 7.5 Hz, 3H); **^{13}C NMR** (75 MHz, DMSO-d6) δ 171.0, 166.1, 152.5, 131.8, 128.8, 127.2, 126.5, 111.2, 14.3, 12.6. Consistent with previous reports in the literature. [1] Substrates **1b-1u** were prepared by the same procedures.

3.2 Para-quinone methide derivatives: 2a-2i, 2j, 5a-5c, 2a-2i, **2j**, **5a-5c** were prepared according to the literature.[2]



Step-1: A solution of phenols (2.14 g, 12 mmol, 1.2 equiv.) and aldehydes (2.36 g, 10 mmol, 1.0 equiv.) in toluene (50 mL) was placed in a Dean-Stark apparatus which was heated to reflux. Piperidine (2.0 equiv.) was added dropwise slowly. The reaction temperature was raised to 140 °C and stirred for 12 h and the reaction mixture was cooled to 120 °C and acetic anhydride was slowly added dropwise to the reaction solution. The stirring was continued for additional 30 min and the solution was poured on ice-water and extracted with CH_2Cl_2 (3 × 50 mL). The organic phases were combined, washed with brine and dried over anhydrous Na_2SO_4. After the solvent was evaporated under reduced pressure, the corresponding products **2a'-2i'** were obtained after flash column chromatography (petroleum ether/ethyl acetate= 100/1).

Step-2: To a solution of **2a'-2i'** (1.0 equiv.) in THF (10 mL/mmol substrate) at 0 °C was added tetrabutylammonium fluoride (1.1 equiv.). The reaction mixture was stirred for 10 min and a saturated NH_4Cl solution was added dropwise to quench the reaction. The resulting solution was extracted with CH_2Cl_2 (3 × 50 mL). The organic phases were combined washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed to give the crude product which was purified by flash column chromatography (petroleum ether/ethyl acetate = 30/1 to 10/1) to afford the desired compounds **2a-1i** and **5c**.[2a]

Substrates **5a**, **5b** were prepared according to the Step-1 described above. [2b]

3.3 Catalysts 4a-4j

Catalysts **4** were prepared according to the reported methods described in the literatures.[3]

3.4 References

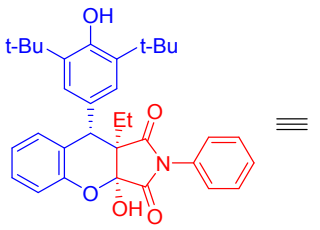
1 (a) Y. Yang, H.-X. Ren, *Org. Lett*. 2017, **19**, 2805-2808; (b) M. A. Tantray, I. Khan, H. Hamid, M. S. Alam, *New J. Chem*., 2016, **40**, 6109-6119.

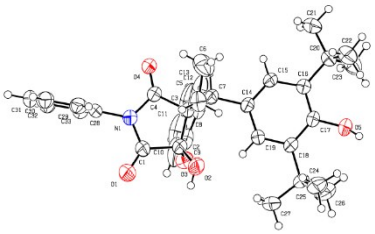
2 (a) K. Zhao, Y. Zhi, *Angew. Chem. Int. Ed.* 2016, **55**, 12104-12108; *Angew. Chem.* 2016, **128**, 12283-12287; (b) L. Roiser, M. Waser, *Org. Lett.* 2017, **19**, 9, 2338–2341.

3 (a) B. Vakulya, S. Varga, A. Csámpai, T. Soós, *Org. Lett.* 2005, **7**, 1967; (b) Q. Zhu, Y. Lu, *Angew. Chem. Int. Ed.* 2010, **49**, 7753-7756; *Angew. Chem.* 2010, **122**, 7919-7922; (c) E. Badiola, B. Fiser, E. Gomez-Bengoa, *J. Am. Chem. Soc*. 2014, **136**, 17869-17881.

4 X-ray crystallographic data

4.1 X-ray crystallographic data of compound 3aa

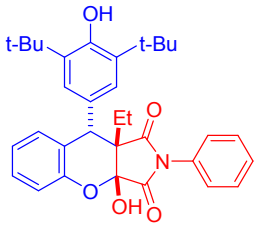




3aa	CCDC 1991270
Identification code	**3aa**
Empirical formula	$C_{33}H_{37}NO_5$
Formula weight	527.63
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	10.2748(4)
b/Å	14.2248(3)
c/Å	10.9525(3)
α/°	90
β/°	115.500(4)
γ/°	90
Volume/Å^3	1444.85(8)
Z	2
ρ_{calc}g/cm^3	1.213
μ/mm^{-1}	0.648
F(000)	564.0
Crystal size/mm^3	0.15 × 0.11 × 0.1

Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.946 to 134.15
Index ranges	-12 ≤ h ≤ 8, -16 ≤ k ≤ 16, -12 ≤ l ≤ 13
Reflections collected	10407
Independent reflections	5148 [R_{int} = 0.0297, R_{sigma} = 0.0395]
Data/restraints/parameters	5148/51/362
Goodness-of-fit on F^2	1.049
Final R indexes [I>=2σ (I)]	R_1 = 0.0436, wR_2 = 0.1149
Final R indexes [all data]	R_1 = 0.0486, wR_2 = 0.1214
Largest diff. peak/hole / e $Å^{-3}$	0.22/-0.15
Flack parameter	-0.05(14)

4.2 X-ray crystallographic data of compound 3aa'



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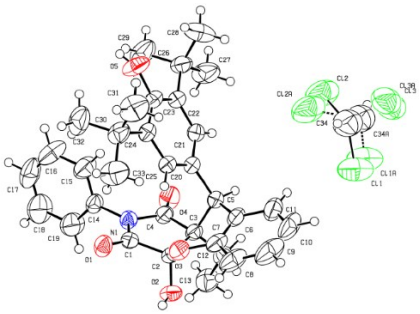


3aa'	CCDC 1991274
Empirical formula	$C_{34}H_{38}Cl_3NO_5$
Formula weight	647.00
Temperature/K	293(2)
Crystal system	triclinic
Space group	P-1
a/Å	10.5380(6)
b/Å	12.9179(10)
c/Å	13.0658(9)
α/°	98.879(6)
β/°	105.364(6)
γ/°	95.484(6)
Volume/$Å^3$	1677.0(2)
Z	2

$\rho_{calc}g/cm^3$	1.281
μ/mm^{-1}	2.802
F(000)	680.0
Crystal size/mm^3	0.22 × 0.14 × 0.1
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.142 to 134.124
Index ranges	-12 ≤ h ≤ 9, -14 ≤ k ≤ 15, -15 ≤ l ≤ 15
Reflections collected	11890
Independent reflections	6004 [R_{int} = 0.0401, R_{sigma} = 0.0539]
Data/restraints/parameters	6004/0/411
Goodness-of-fit on F^2	1.039
Final R indexes [I>=2σ (I)]	R_1 = 0.0716, wR_2 = 0.1933
Final R indexes [all data]	R_1 = 0.0934, wR_2 = 0.2204
Largest diff. peak/hole / e $Å^{-3}$	0.44/-0.47

Single crystal of **3aa** was obtained by slow evaporation from a mixture of ethyl acetate and petroleum ether at 25 °C. ORTEP diagram of compound 3aa, the ellipsoid contour probability levels: 50%.

Single crystal of **3aa'** was obtained by slow evaporation from $CDCl_3$ at 25 °C. ORTEP diagram of compound **3aa'**, the ellipsoid contour probability levels: 50%.

5 Procedures and characterization data

5.1 Typical procedures for the organocatalytic enantioselective [4 + 2] cyclization between hydroxymaleimide 1a and ortho-hydroxyphenyl para-quinone methide 2a

A solution of hydroxymaleimide 1a (21.7 mg, 0.1 mmol, 1 equiv.), para-Quinone Methide 2a (31.0 mg, 0.12 mmol, 1.2 equiv.), and cinchonidine squaramide catalyst **4d** (3 mg, 0.005 mmol, 5% equiv.) in 1,4-dioxane (mL, 0.1 M) was stirred at 25 °C. After **1a** was consumed by TLC (14 h, Rf = 0.1, PE: EA = 20:1), the solvent was evaporated and the mixture was directly purified by flash column chromatography (petroleum ether/ethyl acetate 20/1 to 15/1) to afford products **3aa** (42.8 mg, Rf = 0.2), **3aa'** (9.4 mg, Rf = 0.1). Other reactants were operated by the same procedures. The two diastereomers of each product are separated by chromatographic column.

5.2 Scale-up preparation and representative transformation of product 3aa and control experiments.

5.2.1 Scale-up preparation (Scheme S1 a).

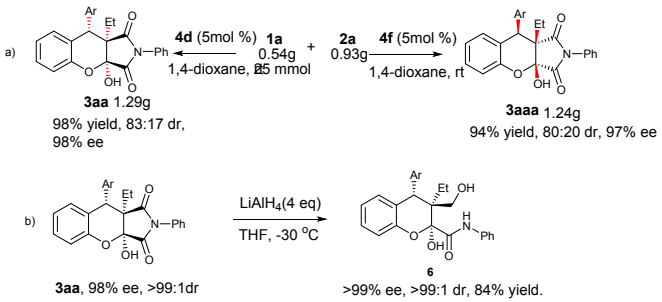
3aa: A solution of hydroxymaleimide **1a** (0.542 g, 2.5 mmol, 1 equiv.), ortho-Hydroxyphenyl substituted para-quinone methides **2a** (0.93 g, 3 mmol, 1.2 equiv.), and catalyst **4d** (75 mg, 2.5 mmol, 0.05 equiv.) in 1,4-dioxane (25 mL, 0.1 M) was stirred at 25 °C. After **1a** was consumed monitored by TLC (26 h, Rf = 0.2, PE: EA = 20:1), the solvent was evaporated and the mixture was directly purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (20:1 to 15:1) to afford product **3aa** (1.29 g, 98% yield, 83:17 dr, 98% ee)

3aaa: A solution of hydroxymaleimide **1a** (0.542 g, 2.5 mmol, 1 equiv.), ortho-Hydroxyphenyl substituted para-quinone methides **2a** (0.93 g, 3 mmol, 1.2 equiv.), and catalyst **4f** (75 mg, 2.5 mmol, 0.05 equiv.) in 1,4-dioxane (25 mL, 0.1 M) was stirred at 25 °C. After **1a** was consumed by TLC (28 h, Rf = 0.2, PE: EA = 20:1), the solvent was evaporated and the mixture was directly purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (20:1 to 15:1) to afford product **3aaa** (1.27 g, 94% yield, 80:20 dr, -97% ee)

5.2.2 Representative transformation (Scheme S1 b)

$LiAlH_4$ (30.4 mg, 0.8 mmol, 4 equiv.) was dissolved in 2 mL of THF and cooled to -30 °C, which was slowly added dropwise to a solution of **3aa** (>99:1 dr, 98% ee) (106 mg, 0.2 mmol, 1eq) in 3 mL of THF also precooled to -30 °C. When **3aa** was consumed monitored by TLC (5 h, Rf = 0.2, PE: EA = 2:1), the reaction was carefully quenched with water (5 mL) at -30 °C and extracted with DCM (10 mL × 3). The organic layer was dried over anhydrous Na_2SO_4 and concentrated. The pure product **6** (89 mg, Rf = 0.2, PE: EA = 2:1) was obtained by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (10:1-5:1).

Scheme S 1 Scale-up preparation and representative transformation of the product **3aa** and control experiments**.**



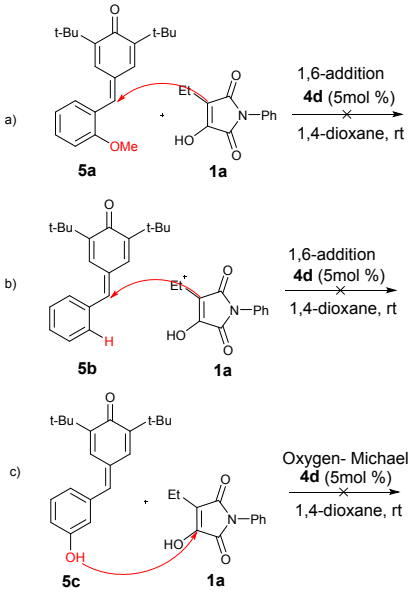
Reaction conditions: yields referred to isolated yields; dr determined by HPLC using crude reaction mixture; ee determined by chiral HPLC, all reactions were completed within 24h, Ar=3,5-di-tert-butyl-4-hydroxyphenyl.

5.3 Reaction mechanism study.

5.3.1 Control experiments

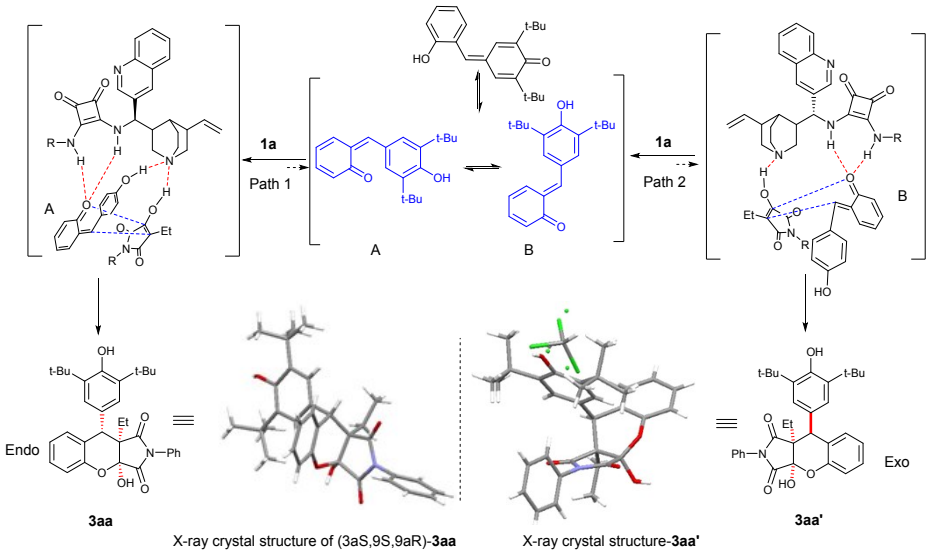
To gain some insight awareness for this new reaction, some control experiments were performed. When hydroxymaleimide **1**a and Me-masked hydroxyphenyl p-QM **5a** are put together under standard conditions, they failed to give 1,6-addition product (Scheme S2 a). We wonder whether the steric hindrance of MeO group caused the reaction failed. **5b** with less steric hindrance was chosen as the substrate, the expected 1,6-addition product (Scheme S2 b) was not observed neither. On the other hand, when changing the position of the hydroxyl group in **2a** from ortho to meta position, the expected oxa-addition product was still not detected (Scheme S1 c).

Scheme S 2 Control experiments.



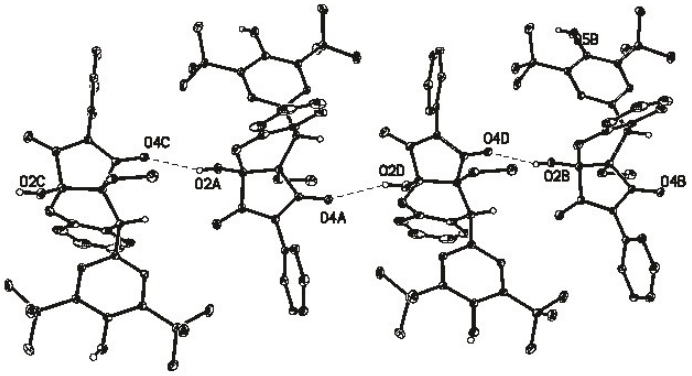
5.3.1 Plausible Diels-Alder reaction mechanism

Scheme S 3 Plausible Diels-Alder reaction mechanism.



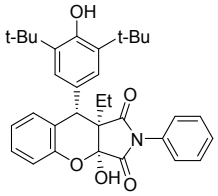
5.3.3 Intermolecular hydrogen bond.

Figure S1 Intermolecular hydrogen bond



5.4 Characterization data

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3aa)



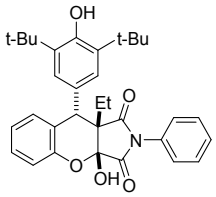
White solid, 99% yield (52.2 mg), 88:12 dr, >99% ee; mp = 214.5-215.6 °C; [α] D^{20} = +405.6 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.6 min, t_{major} = 5.4 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.35 (m, 3H), 7.33 (d, J = 1.2 Hz, 2H), 7.20-7.13 (m, 2H), 7.07-6.94 (m, 4H), 5.14 (s, 1H), 4.99-4.83 (m, 1H), 4.46 (s, 1H), 1.87-1.74 (m, 2H), 1.42 (s, 18H), 1.00 (t, J = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 173.4, 153.1, 150.8, 135.9, 131.1, 129.6, 129.4, 129.1, 128.9, 128.4, 126.3, 126.3, 123.8, 117.8, 98.6, 56.0, 48.3, 34.6, 30.5, 27.4, 9.8;

HRMS(ESI) m/z calcd for $C_{33}H_{37}NO_5Na^+$ $(M+Na)^+$ 550.2564, found 550.2569.

9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3aa')



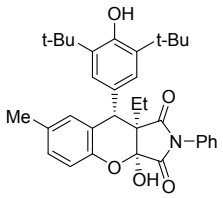
White solid, mp = 196.5-197.8 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.26 (m, 2H), 7.26-7.19 (m, 4H), 7.11-6.99 (m, 3H), 6.54-6.41 (m, 2H), 5.19 (s, 1H), 4.45 (s, 1H), 4.28 (s, 1H), 2.18-2.09 (m, 1H), 1.97-1.89 (m, 1H), 1.30 (s, 18H), 1.01 (t, J = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.8, 172.9, 153.6, 151.5, 136.0, 130.8, 129.0, 128.9, 128.9, 128.8, 128.5, 127.8, 126.6, 124.0, 118.2, 98.2, 56.0, 50.6, 34.5, 30.2, 25.9, 10.2;

HRMS(ESI) m/z calcd for $C_{33}H_{37}NO_5Na^+$ $(M+Na)^+$ 550.2564, found 550.2567.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-7-methyl-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ab)



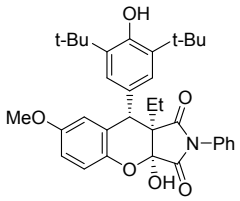
White solid, 95% yield (51.4 mg), 91:9 dr, 98% ee; mp = 226.1-228.1°C; [α] D^{20}= +340.4 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.0 min, t_{major} = 4.6 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.34 (m, 5H), 7.09-7.04 (m, 2H), 6.99-6.91 (m, 3H), 5.16 (s, 1H), 5.09 (s, 1H), 4.40 (s, 1H), 2.23 (s, 3H), 1.84-1.76 (m, 2H), 1.43 (s, 18H), 1.00 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.1, 173.6 (d, J = 7.0 Hz), 153.1, 148.5, 135.8, 133.2, 131.1, 129.9, 129.5, 129.3, 129.1, 128.1, 126.4, 126.3, 117.5, 98.6, 56.1, 48.5, 34.6, 30.5, 27.4, 20.9, 9.9;

HRMS(ESI) m/z calcd for $C_{34}H_{39}NO_5Na^+$ $(M+Na)^+$ 564.2720, found 564.2725.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-7-methoxy-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ac)



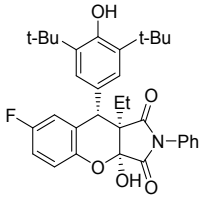
White solid, 99% yield (55.1 mg), 75:25 dr, 96% ee; mp = 204.1-205.8 °C; [α] D^{20}= +261.1 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 5.0 min, t_{major} = 6.2 min);

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51-7.33 (m, 3H), 7.28 (s, 2H), 7.16-6.98 (m, 3H), 6.67-6.43 (m, 2H), 5.12 (s, 1H), 4.79 (s, 1H), 4.39 (s, 1H), 3.73 (s, 3H), 1.82-1.72 (m, 2H), 1.41 (s, 18H), 0.97 (t, J = 7.4 Hz, 3H);

^{13}C NMR (75 MHz, $CDCl_3$) δ 176.9, 173.3, 160.1, 153.0, 151.5, 135.8, 131.0, 130.1, 129.8, 129.4, 129.1, 126.2, 126.2, 120.3, 110.0, 102.9, 98.4, 56.0, 55.5, 47.3, 34.6, 30.5, 27.6, 9.8;

HRMS(ESI) m/z calcd for $C_{34}H_{39}NO_6Na^+$ $(M+Na)^+$ 580.2667, found 580.2673.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-7-fluoro-3a-hydroxy-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ad)



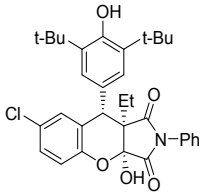
White solid, 99% yield (54.0 mg), 81:19 dr, 98% ee; mp = 224.1-225.7 °C; [α] D^{20}= +334.5 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 6.2 min, t_{major} = 7.8 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.37 (m, 3H), 7.34-7.29 (m, 2H), 7.10-7.04 (m, 2H), 7.01-6.95 (m, 1H), 6.92-6.83 (m, 2H), 5.18 (s, 1H), 5.00 (s, 1H), 4.42 (s, 1H), 1.86-1.75 (m, 2H), 1.42 (s, 18H), 0.99 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.8, 173.2, 158.6 (d, J = 243.0 Hz), 153.3, 146.7, 136.0, 130.9, 130.0 (d, J = 8.1 Hz), 129.4, 129.3, 128.6, 126.3, 126.2, 119.0 (d, J = 8.0 Hz), 115.9 (d, J = 23.6 Hz), 115.6 (d, J = 23.4 Hz), 98.7, 55.7, 48.4, 34.6, 30.5, 27.3, 9.8;

HRMS(ESI) m/z calcd for $C_{33}H_{36}FNO_5Na^+$ (M+Na) $^+$ 568.2469, found 568.2473.

(3aS,9S,9aR)-7-chloro-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ae)



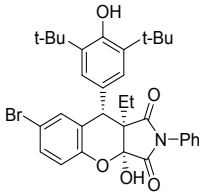
White solid, 98% yield (55.0 mg), 84:16 dr, >99% ee; mp = 243.1-245.0 °C; [α] D^{20}= +345.0 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 97/3, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 7.8 min, t_{major} = 11.8 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.38 (m, 3H), 7.30-7.27 (m, 2H), 7.17-7.07 (m, 4H), 6.97-6.92 (m, 1H), 5.18 (s, 1H), 4.96 (s, 1H), 4.41 (s, 1H), 1.83-1.73 (m, 2H), 1.42 (s, 18H), 0.98 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 173.1, 153.3, 149.3, 136.0, 130.9, 129.7, 129.4, 129.3, 128.9, 128.6, 128.5, 126.2, 126.2, 119.0, 98.6, 55.6, 47.9, 34.6, 30.5, 27.4, 9.7;

HRMS(ESI) m/z calcd for $C_{33}H_{36}ClNO_5Na^+$(M+Na) $^+$ 584.2174, found 584.2178, 586.2161.

(3aS,9S,9aR)-7-bromo-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3af)



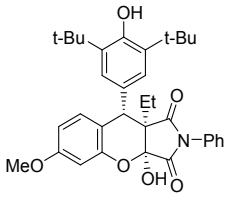
White solid, 94% yield (56.9 mg), 83:17 dr, 99% ee; mp = 234.5-236.1 °C; [α] D^{20}= +297.8 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 97/3, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 7.8 min, t_{major} = 11.8 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.37 (m, 3H), 7.29-7.25 (m, 4H), 7.11-7.06 (m, 2H), 6.91-6.86 (m, 1H), 5.17 (s, 1H), 4.73 (s, 1H), 4.39 (s, 1H), 1.80-1.71 (m, 2H), 1.42 (s, 18H), 0.97 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 172.9, 153.4, 149.9, 136.1, 132.4, 131.9, 130.9, 130.1, 129.4, 129.3, 128.6, 126.2, 126.2, 119.4, 116.1, 98.6, 55.6, 47.7, 34.6, 30.5, 27.4, 9.7;

HRMS(ESI) m/z calcd for $C_{33}H_{36}BrNO_5Na^+$ (M+Na) $^+$ 628.1669, found 628.1673, 632.1709

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-6-methoxy-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ag)



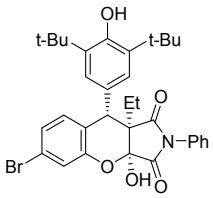
White solid, 94% yield (52.4 mg), 84:16 dr, 96% ee; mp = 203.7-205.1 °C; [α] D^{20}= +292.9 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.8 min, t_{major} = 6.2 min;

^{1}H NMR (300 MHz, DMSO-d6) δ 9.01 (s, 1H), 7.53-7.39 (m, 3H), 7.28 (s, 2H), 7.13-7.04 (m, 1H), 7.01-6.90 (m, 2H), 6.79 (s, 1H), 6.51 (s, 2H), 4.31 (s, 1H), 3.69 (s, 3H), 1.82-1.46 (m, 2H), 1.34 (s, 19H), 0.86 (t, J = 7.3 Hz, 4H);

^{13}C NMR (75 MHz, DMSO-d6) δ 176.6, 172.3, 159.4, 152.5, 151.5, 138.6, 131.0, 130.4, 129.7, 129.4, 129.0, 126.4, 125.7, 120.4, 109.1, 102.3, 99.0, 55.3, 55.0, 46.5, 34.6, 30.5, 26.6, 9.3;

HRMS (ESI) m/z calcd for $C_{34}H_{39}NO_6Na^+$ $(M+Na)^+$ 580.2667, found 580.2673.

(3aS,9S,9aR)-6-bromo-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ah)



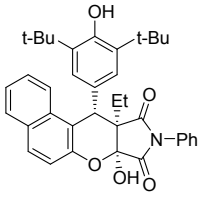
White solid, 99% yield (59.9 mg), 79:21 dr, 99% ee; mp = 223.4-224.7 °C; [α] D^{20}= +274.6 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.1 min, t_{major}= 5.0 min);

^{1}H NMR (300 MHz, DMSO-d6) δ 9.27 (s, 1H), 7.54-7.40 (m, 3H), 7.30 (s, 2H), 7.24-7.10 (m, 3H), 7.05-6.95 (m, 2H), 6.84 (s, 1H), 4.42 (s, 1H), 1.80-1.50 (m, 2H), 1.35 (s, 18H), 0.87 (t, J = 7.3 Hz, 3H);

^{13}C NMR (75 MHz, DMSO-d6) δ 176.3, 171.8, 152.8, 151.6, 138.8, 131.0, 130.8, 129.4, 129.1, 127.7, 126.3, 126.0, 125.8, 120.4, 119.8, 99.5, 54.6, 46.3, 34.6, 30.5, 26.5, 9.2;

HRMS (ESI) m/z calcd for $C_{33}H_{36}BrNO_5Na^+$ $(M+Na)^+$ 628.1669, found 628.1673, 632.1712.

(7aR,10aS,11R)-11-(3,5-di-tert-butyl-4-hydroxyphenyl)-10a-ethyl-7a-hydroxy-9-phenyl-10a,11-dihydrobenzo [5,6] chromeno [2,3-c] pyrrole-8,10(7aH,9H)-dione (3ai)



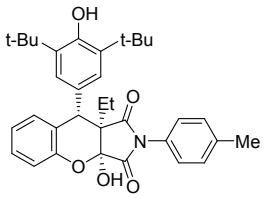
White solid, 85% yield (49.0 mg), >99:1 dr, -99% ee; mp = 238.1-239.7 °C; [α] D^{20}= -148.1 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 5.9 min, t_{major} = 9.4 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.90 (m, 1H), 7.89-7.77 (m, 2H), 7.57-7.49 (m, 1H), 7.48-7.44 (m, 1H), 7.44-7.39 (m, 1H), 7.30-7.27 (m, 2H), 7.25 (s, 1H), 7.12 (s, 2H), 6.52-6.39 (m, 2H), 5.15 (s, 1H), 4.96 (s, 1H), 4.24 (s, 1H), 2.28-2.13 (m, 1H), 2.05-1.94 (m, 1H), 1.28 (s, 18H), 0.86 (t, J = 7.5 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.0, 172.9, 153.7, 149.3, 136.1, 131.1, 130.9, 130.8, 129.4, 129.0, 128.9, 128.9, 127.8, 127.3, 126.6, 124.6, 122.2, 120.4, 118.7, 97.8, 55.7, 46.0, 34.4, 30.1, 25.8, 10.2;

HRMS(ESI) m/z calcd for $C_{37}H_{39}NO_5Na^+$ $(M+Na)^+$ 600.2720, found 600.2724.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-(p-tolyl)-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ba)



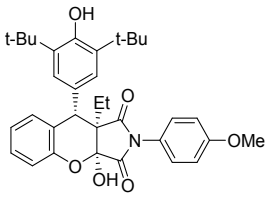
White solid, 99% yield (53.5 mg), 85:15 dr, 98% ee; mp = 179.7-181.5 °C; [α] D^{20}= +351.4 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.6 min, t_{major} = 6.6 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.30 (m, 2H), 7.21-7.13 (m, 4H), 7.02-6.93 (m, 2H), 6.93-6.87 (m, 2H), 5.13 (s, 1H), 4.84-4.58 (m, 1H), 4.44 (s, 1H), 2.35 (s, 3H), 1.83-1.74 (m, 2H), 1.42 (s, 18H), 0.98 (t, J = 7.3 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 173.4, 153.1, 150.8, 139.3, 135.9, 130.0, 129.6, 129.4, 128.8, 128.4, 126.3, 126.0, 123.8, 117.8, 98.6, 56.0, 48.3, 34.6, 30.5, 27.4, 21.3, 9.8;

HRMS(ESI) m/z calcd for $C_{34}H_{39}NO_5Na^+$ $(M+Na)^+$ 564.2720, found 564.2725.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-(4-methoxyphenyl)-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ca)



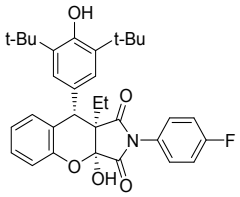
White solid, 95% yield (53.0 mg), 85:15 dr, >99% ee; mp = 178.6-179.7 °C; [α] D^{20}= +328.3 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 6.2 min, t_{major} = 8.6 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.30 (m, 2H), 7.19-7.11 (m, 2H), 7.02-6.88 (m, 6H), 5.13 (s, 1H), 4.82-4.69 (m, 1H), 4.43 (s, 1H), 3.79 (s, 3H), 1.84-1.72 (m, 2H), 1.42 (s, 18H), 0.97 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.2, 173.5, 160.0, 153.1, 150.8, 135.9, 129.6, 129.4, 128.8, 128.5, 127.5, 126.3, 123.8, 123.6, 117.7, 114.7, 98.6, 56.0, 55.6, 48.3, 34.6, 30.5, 27.4, 9.8;

HRMS(ESI) m/z calcd for $C_{34}H_{39}NO_6Na^+$ (M+Na) $^+$ 580.2669, found 580.2672.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-2-(4-fluorophenyl)-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3da)



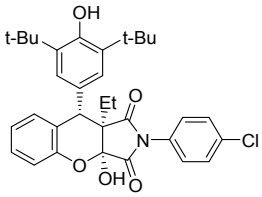
White solid, 94% yield (51.2 mg), 95:5 dr, 98% ee; mp = 199.5-200.6 °C; [α] D^{20}= +353.9 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.4 min, t_{major} = 5.6 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.29 (m, 2H), 7.19-6.95 (m, 8H), 5.14 (s, 1H), 4.97 (s, 1H), 4.45 (s, 1H), 1.84-1.75 (m, 2H), 1.42 (s, 18H), 0.98 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 173.3, 162.6 (d, J = 249.5 Hz), 153.1, 150.7, 135.9, 129.6, 129.2, 128.9, 128.4, 128.2 (d, J = 8.8 Hz), 126.9 (d, J = 3.3 Hz), 126.3, 123.9, 117.8, 116.4 (d, J = 23.0 Hz), 98.6, 56.1, 48.3, 34.6, 30.5, 27.4, 9.8;

HRMS(ESI) m/z calcd for $C_{33}H_{36}FNO_5Na^+$ (M+Na) $^+$ 568.2469, found 568.2473.

(3aS,9S,9aR)-2-(4-chlorophenyl)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ea)



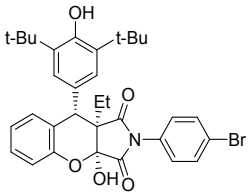
White solid, 98% yield (55.0 mg), 78:22 dr, 98% ee; mp = 177.5-179.2 °C; [α] D^{20}= +315.6 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.6 min, t_{major} = 6.3 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.35 (m, 2H), 7.32-7.29 (m, 2H), 7.19-7.13 (m, 2H), 7.03-6.97 (m, 4H), 5.14 (s, 1H), 4.76 (s, 1H), 4.44 (s, 1H), 1.85-1.74 (m, 2H), 1.41 (s, 18H), 0.96 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.7, 173.1, 153.1, 150.7, 135.9, 135.0, 129.6, 129.5, 129.2, 128.9, 128.3, 127.5, 126.3, 123.9, 117.7, 98.5, 56.1, 48.2, 34.6, 30.5, 27.4, 9.8;

HRMS(ESI) m/z calcd for $C_{33}H_{36}ClNO_5Na^+$ (M+Na) + 584.2174, found 584.2178, 586.2161.

(3aS,9S,9aR)-2-(4-bromophenyl)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3fa)



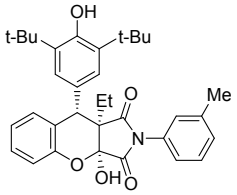
White solid, 95% yield (57.4 mg), 78:22 dr, 98% ee; mp = 212.3-213.225 °C; [α] D^{20}= +267.8 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 97/3, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 12.3 min, t_{major} = 21.9 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.50 (m, 2H), 7.30 (s, 2H), 7.19-7.12 (m, 2H), 7.02-6.92 (m, 4H), 5.14 (s, 1H), 4.71 (s, 1H), 4.43 (s, 1H), 1.84-1.73 (m, 2H), 1.41 (s, 18H), 0.96 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.7, 173.0, 153.1, 150.7, 135.9, 132.6, 130.0, 129.6, 129.1, 128.9, 128.2, 127.7, 126.3, 123.9, 123.1, 117.7, 98.5, 56.1, 48.2, 34.6, 30.5, 27.4, 9.8;

HRMS(ESI) m/z calcd for $C_{33}H_{36}BrNO_5Na^+$ $(M+Na)^+$ 628.1669, found 628.1674, 630.1653.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-(m-tolyl)-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ga)



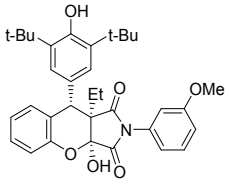
White solid, >99% yield (53.6 mg), 84:16 dr, >99% ee; mp = 200.3-201.3 °C; [α] D^{20} = +349.2 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 3.9 min, t_{major} = 4.4 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.27 (m, 3H), 7.20-7.14 (m, 3H), 7.03-6.94 (m, 2H), 6.85-6.77 (m, 2H), 5.13 (s, 1H), 4.89 (s, 1H), 4.45 (s, 1H), 2.34 (s, 3H), 1.86-1.75 (m, 2H), 1.42 (s, 18H), 0.99 (t, J = 7.5 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 173.5, 153.1, 150.8, 139.5, 135.9, 131.0, 130.0, 129.6, 129.4, 129.2, 128.8, 128.4, 126.8, 126.3, 123.8, 123.4, 117.8, 98.6, 56.0, 48.3, 34.6, 30.5, 27.4, 21.4, 9.8;

HRMS(ESI) m/z calcd for $C_{34}H_{39}NO_5Na^+$ $(M+Na)^+$ 564.2720, found 564.2724.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-(3-methoxyphenyl)-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ha)



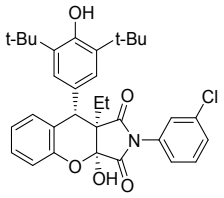
White solid, 96% yield (53.5 mg), 81:19 dr, 98% ee; mp = 171.1-172.6 °C; [α] D^{20} = +331.4 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 97/3, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.6 min, t_{major} = 5.6 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.28 (m, 3H), 7.19-7.13 (m, 2H), 7.02-6.89 (m, 3H), 6.65-6.59 (m, 1H), 6.56-6.50 (m, 1H), 5.13 (s, 1H), 4.81 (s, 1H), 4.44 (s, 1H), 3.76 (s, 3H), 1.84-1.75 (m, 2H), 1.42 (s, 18H), 0.98 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 173.3, 160.2, 153.1, 150.8, 135.9, 132.0, 130.1, 129.6, 129.3, 128.8, 128.4, 126.3, 123.8, 118.5, 117.8, 114.8, 112.3, 98.6, 56.1, 55.6, 48.4, 34.6, 30.5, 27.3, 9.8;

HRMS(ESI) m/z calcd for $C_{34}H_{39}NO_6Na^+$ $(M+Na)^+$ 580.2669, found 580.2674.

(3aS,9S,9aR)-2-(3-chlorophenyl)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ia)



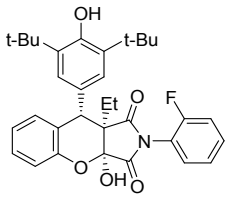
White solid, 94% yield (52.8 mg), 78:22 dr, 98% ee; mp = 204.1-205.9 °C; [α] D^{20} = +328.7 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.0 min, t_{major} = 4.4 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.29 (m, 4H), 7.19-7.14 (m, 2H), 7.11-7.06 (m, 1H), 7.03-6.94 (m, 3H), 5.14 (s, 1H), 4.68 (s, 1H), 4.44 (s, 1H), 1.85-1.74 (m, 2H), 1.41 (s, 18H), 0.97 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 172.9, 153.2, 150.6, 135.9, 135.0, 132.1, 130.3, 129.6, 129.4, 129.2, 129.0, 128.2, 126.5, 126.3, 124.4, 124.0, 117.7, 98.5, 56.1, 48.1, 34.6, 30.5, 27.4, 9.8;

HRMS(ESI) m/z calcd for $C_{33}H_{36}ClNO_5Na^+$ $(M+Na)^+$ 584.2174, found 584.2174, 586.2162.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-2-(2-fluorophenyl)-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ja)



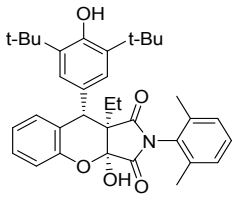
White solid, 96% yield (52.5 mg), 99:1 dr, >99% ee; mp = 220.7-222.5 °C; [α] D^{20} = +342.3 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.4 min, t_{major} = 5.7 min);

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.32 (m, 3H), 7.25-7.14 (m, 4H), 7.14-6.87 (m, 3H), 5.21 (s, 1H), 5.16 (s, 1H), 4.46 (s, 1H), 1.87-1.79 (m, 2H), 1.42 (s, 18H), 1.04 (t, J = 7.4 Hz, 3H);

^{13}C NMR (75 MHz, $CDCl_3$) δ 176.2, 172.9, 157.3 (d, J = 254.6 Hz), 153.1, 150.7, 135.8, 131.4 (d, J = 7.6 Hz), 129.4, 129.2, 129.0, 128.8, 126.3, 124.7, 123.9, 118.8 (d, J = 13.9 Hz), 117.9, 116.9 (d, J = 19.1 Hz), 98.9, 56.6, 48.4, 34.6, 30.5, 27.2, 9.7;

HRMS(ESI) m/z calcd for $C_{33}H_{36}FNO_5Na^+$ $(M+Na)^+$ 568.2469, found 568.2473.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-2-(2,6-dimethylphenyl)-9a-ethyl-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ka)



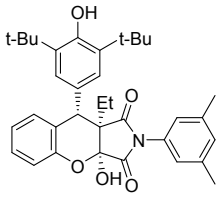
White solid, 96% yield (53.3 mg), 96:4 dr, >99% ee; mp = 197.1-198.9 °C; [α] D^{20}= +284.0 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.1 min, t_{major} = 7.1 min);

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (s, 2H), 7.23-7.06 (m, 4H), 7.04-6.90 (m, 3H), 5.13 (s, 1H), 4.87 (s, 1H), 4.52 (s, 1H), 2.13 (s, 3H), 1.92-1.71 (m, 2H), 1.42 (s, 18H), 1.33 (s, 3H), 1.07 (t, J = 7.4 Hz, 3H);

^{13}C NMR (75 MHz, $CDCl_3$) δ 176.4, 173.2, 153.1, 150.8, 136.1, 135.9, 135.7, 129.7, 129.7, 129.5, 128.8, 128.8, 128.7, 128.5, 126.1, 123.9, 118.3, 99.2, 56.2, 47.0, 34.6, 30.5, 26.3, 18.3, 16.9, 9.4;

HRMS(ESI) m/z calcd for $C_{35}H_{41}NO_5Na^+$ $(M+Na)^+$ 578.2876, found 578.2880.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-2-(3,5-dimethylphenyl)-9a-ethyl-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3la)



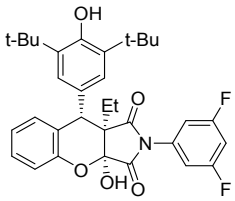
White solid, >99% yield (53.3 mg), 84:16 dr, >99% ee; mp = 164.4-165.7 °C; [α] D^{20} = +331.6 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 3.8 min, t_{major} = 3.8);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.29 (m, 2H), 7.20-7.14 (m, 2H), 7.03-6.95 (m, 3H), 6.59 (s, 2H), 5.13 (s, 1H), 4.73 (s, 1H), 4.43 (s, 1H), 2.29 (s, 6H), 1.84-1.73 (m, 2H), 1.42 (s, 18H), 0.99 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.1, 173.5, 153.1, 150.8, 139.3, 135.9, 131.1, 130.8, 129.6, 129.4, 128.8, 128.4, 126.3, 124.0, 123.8, 117.8, 98.5, 56.0, 48.3, 34.6, 30.5, 27.4, 21.3, 9.9;

HRMS(ESI) m/z calcd for $C_{35}H_{41}NO_5Na^+$ $(M+Na)^+$ 578.2876, found 578.2880.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-2-(3,5-difluorophenyl)-9a-ethyl-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ma)



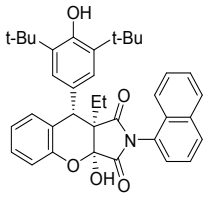
White solid, 93% yield (52.3 mg), 72:28 dr, >99% ee; mp = 129.1-130.7 °C; [α] D^{20} = +253.3 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 3.4 min, t_{major} = 3.8 min);

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (s, 2H), 7.22-7.10 (m, 2H), 7.05-6.94 (m, 2H), 6.90-6.79 (m, 1H), 6.72 (dd, J = 7.2, 2.2 Hz, 2H), 5.15 (s, 1H), 4.92 (s, 1H), 4.44 (s, 1H), 1.84-1.75 (m, 2H), 1.41 (s, 18H), 0.97 (d, J = 7.4 Hz, 3H);

^{13}C NMR (75 MHz, $CDCl_3$) δ 176.3, 172.7, 162.9 (dd, J = 250.1, 13.7 Hz), 153.2, 150.5, 135.9, 129.6, 129.0, 129.0, 128.0, 126.3, 124.0, 117.7, 109.8 (d, J = 27.9 Hz), 109.8 (d, J = 9.2 Hz), 104.8 (t, J = 25.2 Hz), 98.4, 56.1, 48.1, 34.6, 30.5, 27.4, 9.8;

HRMS(ESI) m/z calcd for $C_{33}H_{35}F_2NO_5Na^+$ $(M+Na)^+$ 586.2375, found 586.2377.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-2-(naphthalen-1-yl)-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3na)



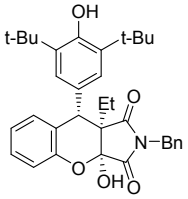
White solid, 99% yield (57.1 mg), 75:1 dr, 95% ee; mp = 207.9-208.3 °C; [α] D^{20} = +252.6 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.7 min, t_{major} = 6.0 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.82 (m, 2H), 7.54-7.49 (m, 1H), 7.45-7.39 (m, 3H), 7.38-7.26 (m, 3H), 7.20-7.08 (m, 3H), 6.09 (dd, J = 8.6, 1.1 Hz, 1H), 5.26 (s, 1H), 5.14 (s, 1H), 4.51 (s, 1H), 1.97-1.81 (m, 2H), 1.40 (s, 18H), 1.10 (t, J = 7.4 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.5, 174.1, 153.1, 151.1, 136.0, 134.4, 130.4, 129.8, 129.5, 129.3, 129.1, 129.0, 128.4, 127.9, 127.4, 126.6, 126.3, 125.9, 125.2, 124.2, 121.9, 118.8, 99.3, 56.9, 49.3, 34.6, 30.5, 27.4, 10.1;

HRMS(ESI) m/z calcd for $C_{37}H_{39}NO_5Na^+$ $(M+Na)^+$ 600.2720, found 600.2722.

(3aS,9S,9aR)-2-benzyl-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3oa)



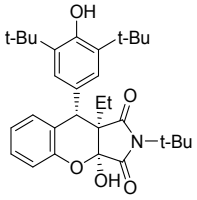
White solid, 99% yield (53.6 mg), 88:12 dr, 95% ee; mp = 208.3 209.1 °C; [α] D^{20} = +118.6 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 5.1 min, t_{major} = 5.5 min);

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (s, 2H), 7.24-7.02 (m, 2H), 7.00-6.79 (m, 2H), 5.11 (s, 1H), 4.80 (s, 1H), 4.71-4.51 (m, 2H), 4.37 (s, 1H), 1.74-1.63 (m, 2H), 1.40 (s, 20H), 0.72 (t, J = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 177.76, 173.94, 153.02, 150.68, 135.84, 134.64, 129.50, 129.29, 128.72, 128.66, 128.54, 126.16, 123.77, 118.07, 98.68, 77.58, 77.16, 76.74, 56.12, 48.49, 42.50, 34.56, 30.49, 27.03, 9.53.

HRMS(ESI) m/z calcd for $C_{34}H_{39}NO_5Na^+$ $(M+Na)^+$ 564.2720, found 564.2725.

(3aS,9S,9aR)-2-(tert-butyl)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-ethyl-3a-hydroxy-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3pa)



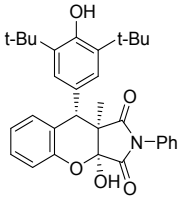
White solid, 96% yield (48.7 mg), 75:25 dr, 93% ee; mp = 187.1-188.8 °C; [α] D^{20} = +235.5 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 3.8 min, t_{major}= 5.5 min);

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 (s, 2H), 7.16-7.06 (m, 2H), 6.98-6.82 (m, 2H), 5.10 (s, 1H), 4.64 (s, 1H), 4.27 (s, 1H), 1.65-1.60 (m, 2H), 1.40 (s, 18H), 1.38 (s, 9H), 0.82 (t, J = 7.4 Hz, 3H);

^{13}C NMR (75 MHz, $CDCl_3$) δ 178.7, 175.3, 152.9, 151.1, 135.7, 129.6, 129.4, 128.6, 128.5, 126.3, 123.4, 117.6, 97.7, 59.3, 55.3, 48.8, 34.5, 30.5, 28.1, 27.2, 9.7;

HRMS(ESI) m/z calcd for $C_{31}H_{41}NO_5Na^+$ $(M+Na)^+$ 530.2877, found 530.2880.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-3a-hydroxy-9a-methyl-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3qa)



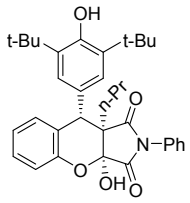
White solid, 96% yield (49.3 mg), 79:21 dr, 98% ee; mp = 199.6-201.4 °C; [α] D^{20} = +302.3 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AS-H, n-hexane/isopropanol = 97/3, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.5 min, t_{major} = 18.0 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.32 (m, 3H), 7.26 (s, 2H), 7.21-7.13 (m, 2H), 7.09-6.92 (m, 4H), 5.14 (s, 1H), 4.45 (s, 1H), 1.42 (s, 18H), 1.33 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 172.9, 153.1, 150.9, 135.9, 131.1, 129.7, 129.3, 129.2, 129.1, 128.9, 128.0, 126.4, 126.2, 123.9, 117.8, 98.8, 52.3, 48.1, 34.6, 30.5, 19.6;

HRMS(ESI) m/z calcd for $C_{32}H_{35}NO_5Na^+$ $(M+Na)^+$ 536.2407, found 536.2411.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-3a-hydroxy-2-phenyl-9a-propyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ra)



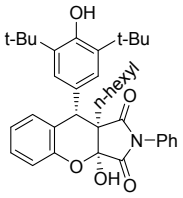
White solid, 97% yield (52.5 mg), 95:5 dr, 99% ee; mp = 182.1-183.6 °C; [α] D^{20} = +337.9 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 4.3 min, t_{major} = 6.1 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.36 (m, 3H), 7.33-7.29 (m, 2H), 7.19-7.13 (m, 2H), 7.05-6.95 (m, 4H), 5.15 (s, 1H), 4.95 (s, 1H), 4.48 (s, 1H), 1.75-1.63 (m, 2H), 1.42 (s, 18H), 1.39-1.25 (m, 2H), 0.86 (t, J = 7.1 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.0, 173.4, 153.1, 150.8, 135.8, 131.1, 129.6, 129.3, 129.2, 129.1, 128.9, 128.4, 126.3, 126.2, 123.8, 117.8, 98.8, 55.8, 48.2, 36.1, 34.6, 30.5, 18.6, 14.5;

HRMS(ESI) m/z calcd for $C_{34}H_{39}NO_5Na^+$ $(M+Na)^+$ 564.2720, found 564.2724.

(3aS,9S,9aR)-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-9a-hexyl-3a-hydroxy-2-phenyl-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3sa)



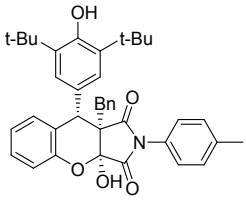
White solid, 95% yield (55.4 mg), 79:21 dr, 99% ee; mp = 178.7-180.5 °C; [α] D^{20} = +313.2 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 90/10, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 5.4 min, t_{major} = 8.8 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.36 (m, 3H), 7.32-7.29 (m, 2H), 7.19-7.14 (m, 2H), 7.05-6.95 (m, 4H), 5.14 (s, 1H), 4.85 (s, 1H), 4.48 (s, 1H), 1.72-1.68 (m, 2H), 1.42 (s, 18H), 1.28-1.15 (m, 8H), 0.84 (t, J = 6.6 Hz, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 176.9, 173.3, 153.1, 150.8, 135.9, 131.1, 129.6, 129.4, 129.2, 129.1, 128.8, 128.2, 126.4, 126.2, 123.8, 117.7, 98.7, 55.6, 47.9, 34.6, 34.0, 31.6, 30.5, 29.7, 25.0, 22.6, 14.1;

HRMS(ESI) m/z calcd for $C_{37}H_{45}NO_5Na^+$ $(M+Na)^+$ 606.3189, found 606.3193.

(3aS,9S,9aR)-9a-benzyl-9-(3,5-di-tert-butyl-4-hydroxyphenyl)-3a-hydroxy-2-(p-tolyl)-9,9a-dihydrochromeno[2,3-c] pyrrole-1,3(2H,3aH)-dione (3ta)



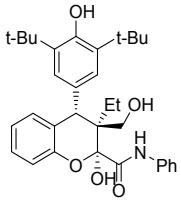
White solid, 98% yield (59.1 mg), 99:1 dr, 98% ee; mp = 237.1-238.1 °C; [α] D^{20} = +423.3 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak AD-H, n-hexane/isopropanol = 97/3, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 5.4 min, t_{major} = 9.1 min);

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (s, 2H), 7.30-7.27 (m, 2H), 7.23-7.14 (m, 5H), 7.04-6.96 (m, 4H), 6.24-6.20 (m, 2H), 5.17 (s, 1H), 4.91 (s, 1H), 4.62 (s, 1H), 3.17 (d, J = 13.4 Hz, 1H), 2.92 (d, J = 13.5 Hz, 1H), 2.27 (s, 3H), 1.67 (s, 1H), 1.44 (s, 18H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.9, 172.5, 153.2, 150.7, 139.1, 136.0, 135.6, 131.0, 129.6, 129.1, 128.9, 128.6, 127.9, 127.5, 126.6, 126.1, 123.7, 117.5, 98.2, 57.5, 49.1, 40.0, 34.6, 30.6, 21.3;

HRMS(ESI) m/z calcd for $C_{39}H_{41}NO_5Na^+$ (M+Na) $^+$ 626.2876, found 626.2879.

2,6-di-tert-butyl-4-((3aS,9R,9aS)-9a-ethyl-2-phenyl-1,2,3,3a,9,9a-hexahydrochromeno[2,3-c] pyrrol-9-yl) phenol (6)



White solid, 91% yield (48.2 mg), >99:1 dr, 99% ee; mp = 121.4-123.2 °C; [α] D^{20} = -90.4 (c 0.50, CH_2Cl_2); the ee was determined by HPLC (Chiralpak IC, n-hexane/isopropanol = 97/3, flow rate 1.0 mL/min, λ = 254 nm, t_{minor} = 8.3 min, t_{major} = 9.2 min);

^{1}H NMR (400 MHz, DMSO-d6) δ 9.29 (s, 1H), 8.49 (d, J = 7.8 Hz, 1H), 7.59 (d, J = 8.0 Hz, 2H), 7.38-7.22 (m, 4H), 7.21-7.11 (m, 1H), 7.00-6.88 (m, 1H), 6.81-6.66 (m, 3H), 5.73 (d, J = 5.1 Hz, 1H), 5.19 (s, 1H), 5.07 (dd, J = 11.4, 6.6 Hz, 1H), 4.31 (t, J = 5.9 Hz, 1H), 1.95 – 1.80 (m, 2H), 1.59 – 1.28 (m, 1H), 1.22 (s, 20H), 0.78 (t, J = 7.3 Hz, 3H).

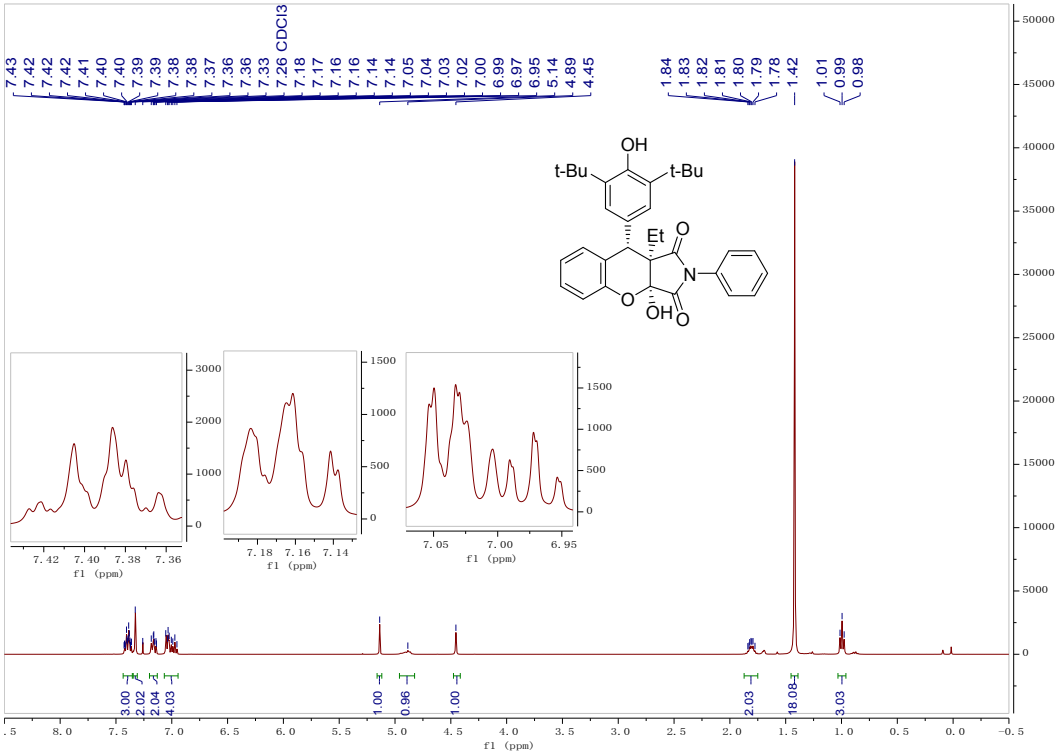
^{13}C NMR (100 MHz, DMSO-d6) δ 176.1, 154.7, 152.1, 138.7, 138.5, 133.2, 131.1, 129.8, 128.2, 126.8, 126.3, 125.1, 122.4, 118.5, 114.9, 82.1, 70.2, 54.4, 34.4, 30.5, 30.3, 30.2, 7.6.

HRMS(ESI) m/z calcd for $C_{33}H_{41}NO_5Na^+$ (M+Na) $^+$ 554.2876, found 554.2879.

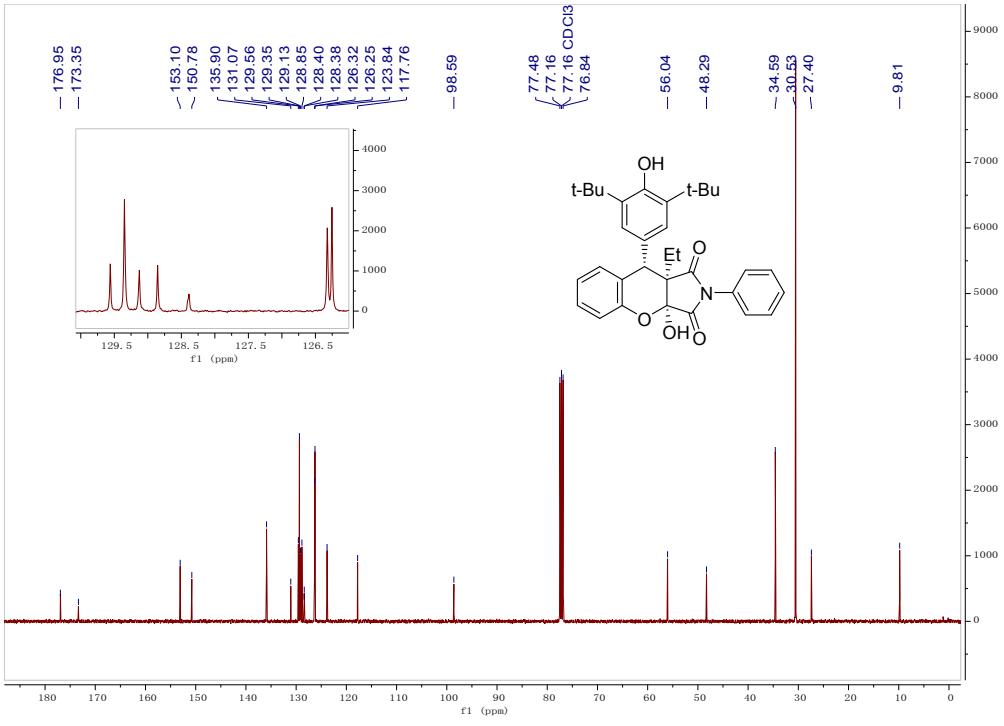
6 NMR, HPLC and HRMS spectra of products

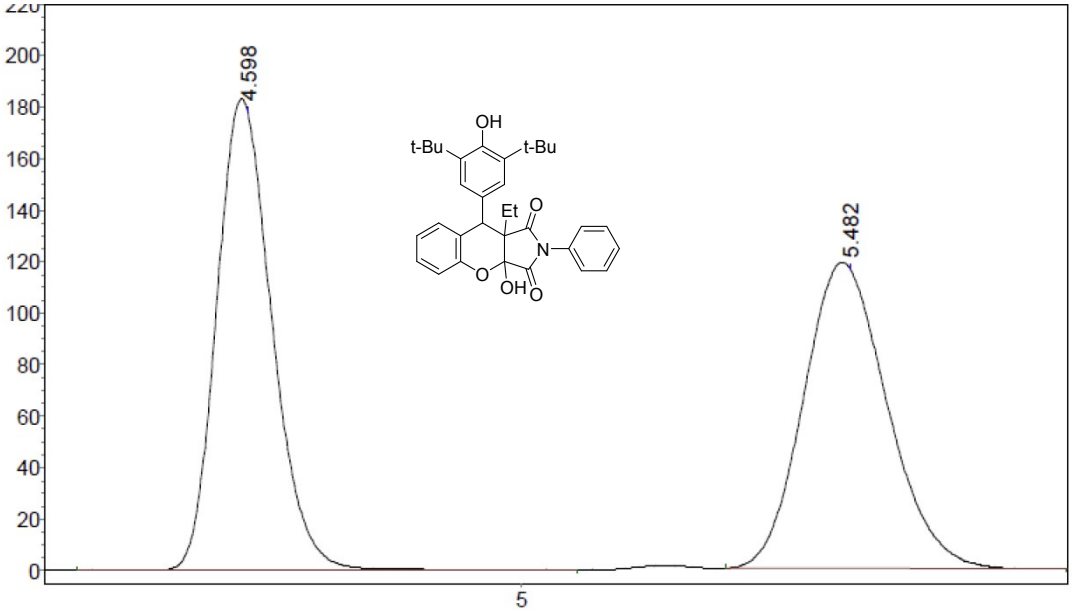
3aa

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

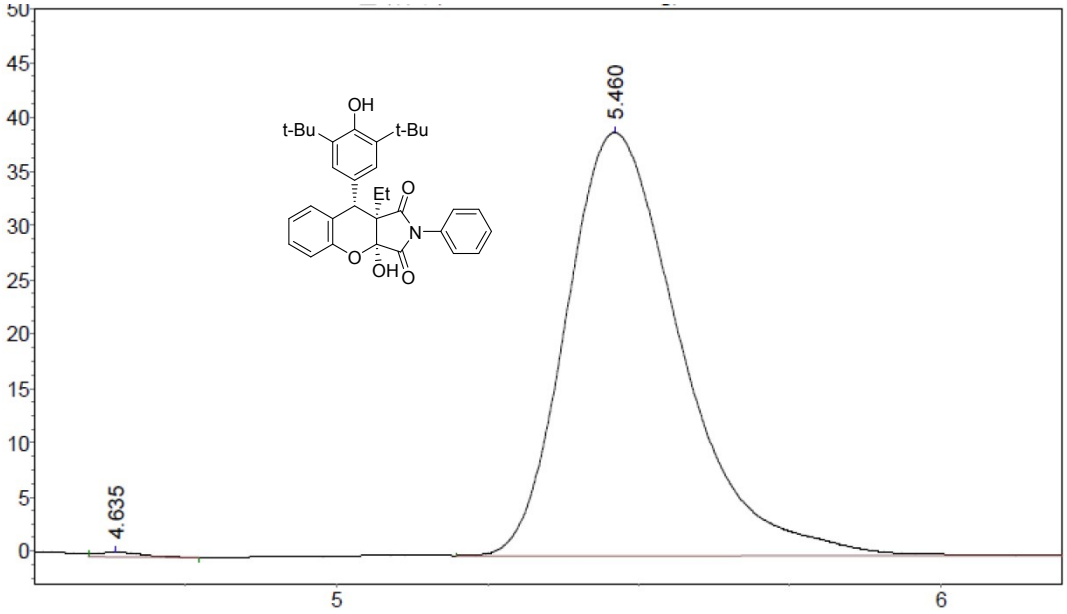


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.598	181363.234	1025639.500	50.1730
2	5.482	118320.695	1018568.438	49.8270

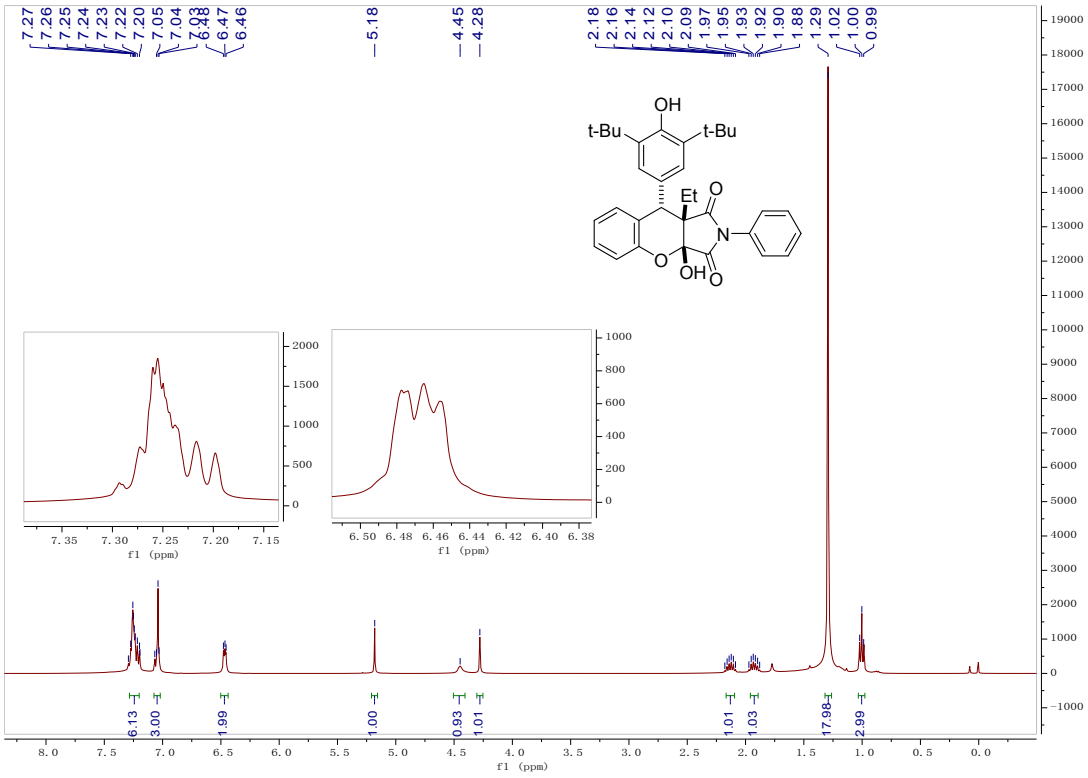


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.635	474.714	2287.540	0.4434
2	5.460	39019.074	513592.813	99.5566

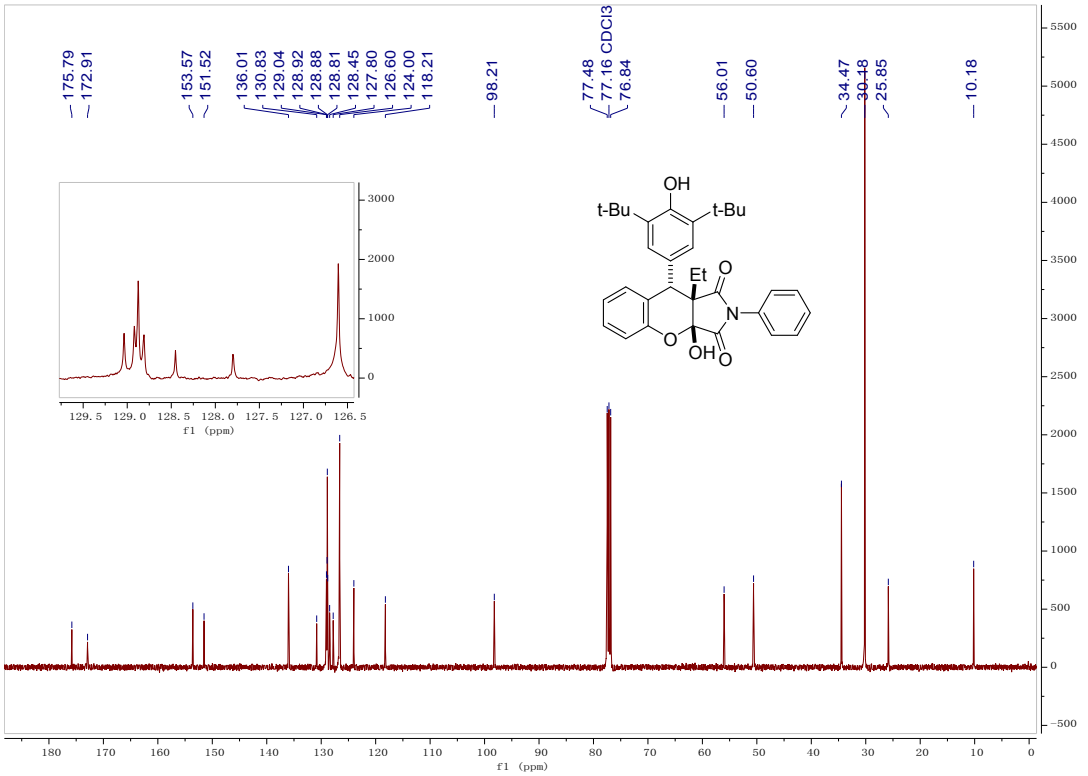
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.6 min, t_{major} = 5.4 min.

3aa'

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

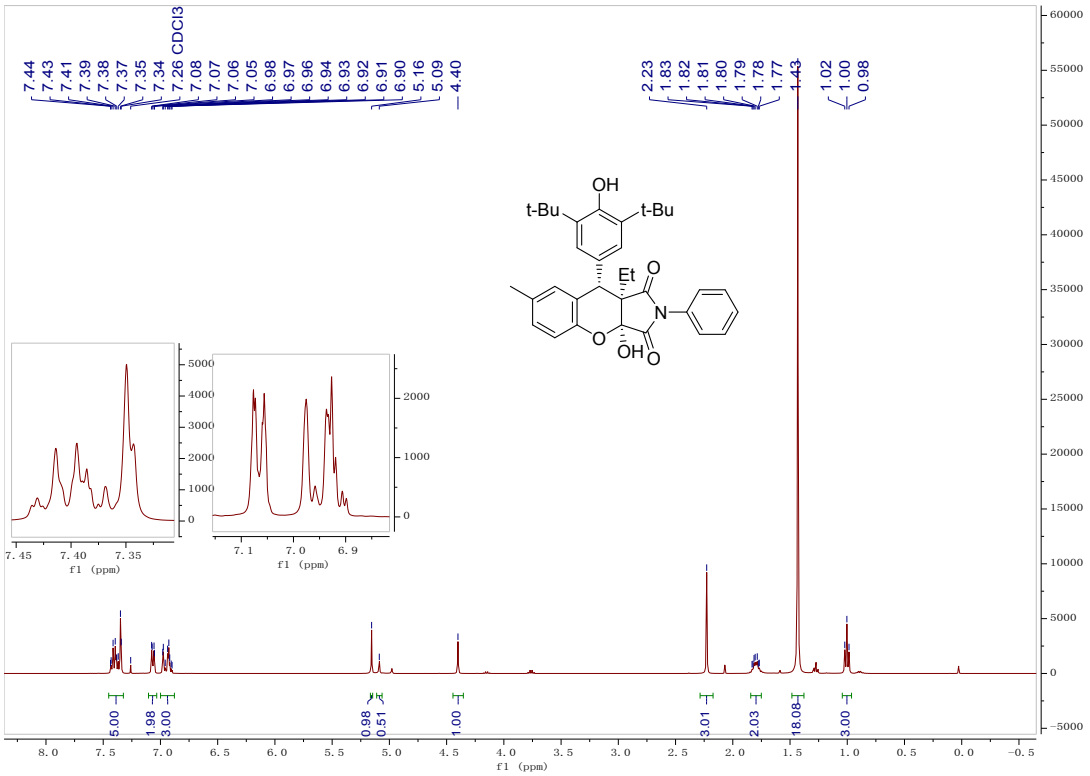


^{13}C NMR (100 MHz, $CDCl_3$)

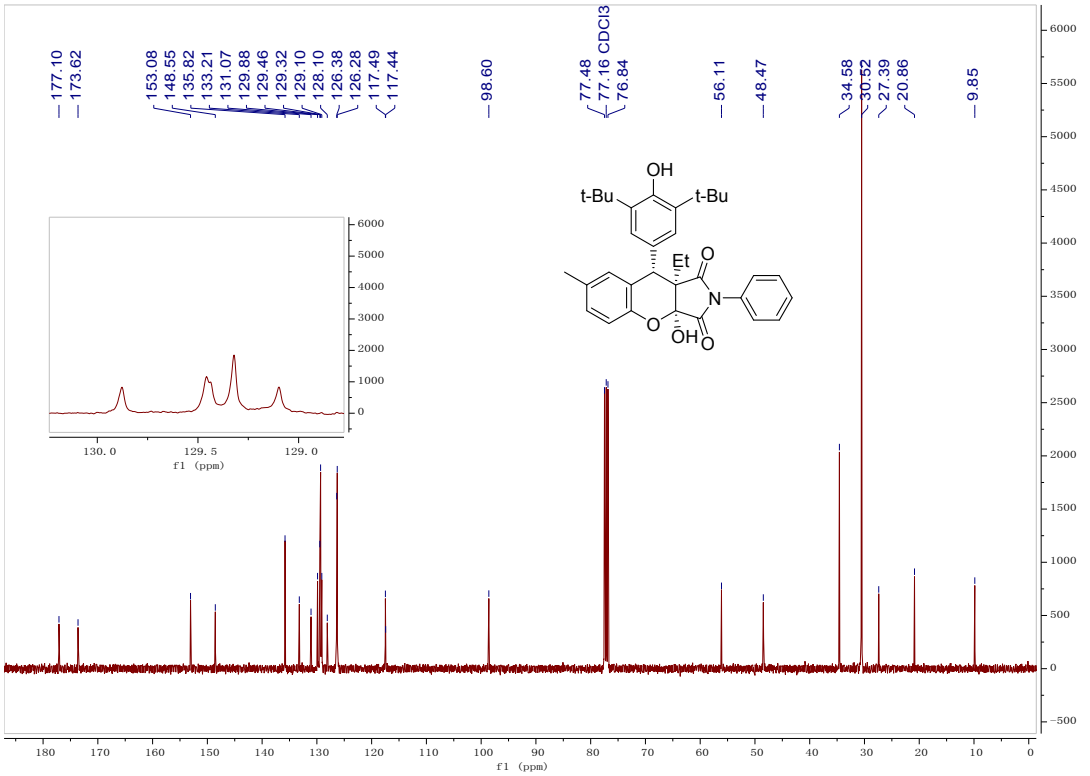


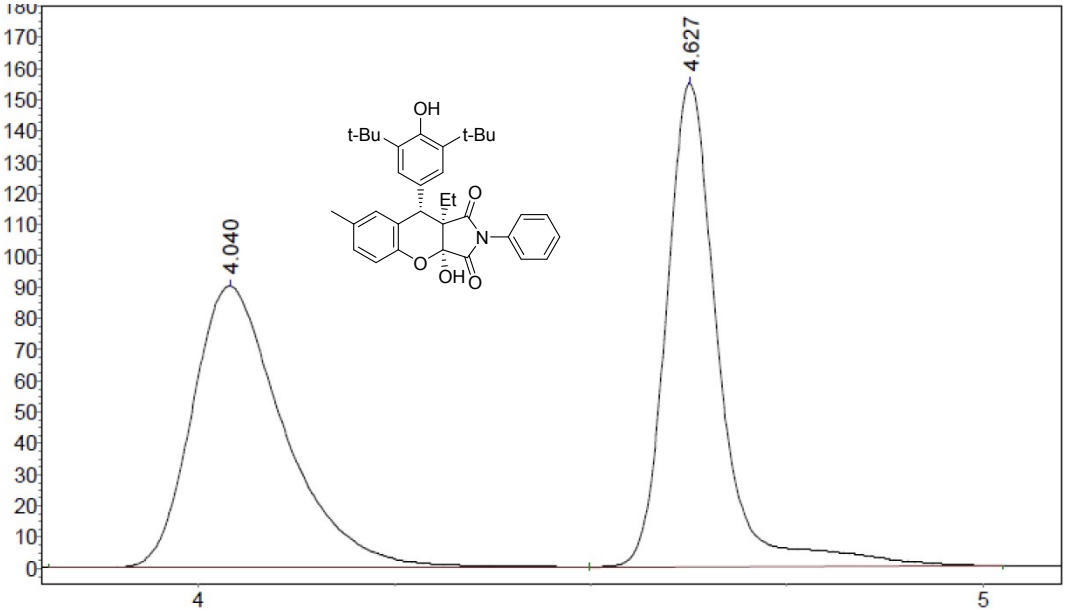
3ab

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

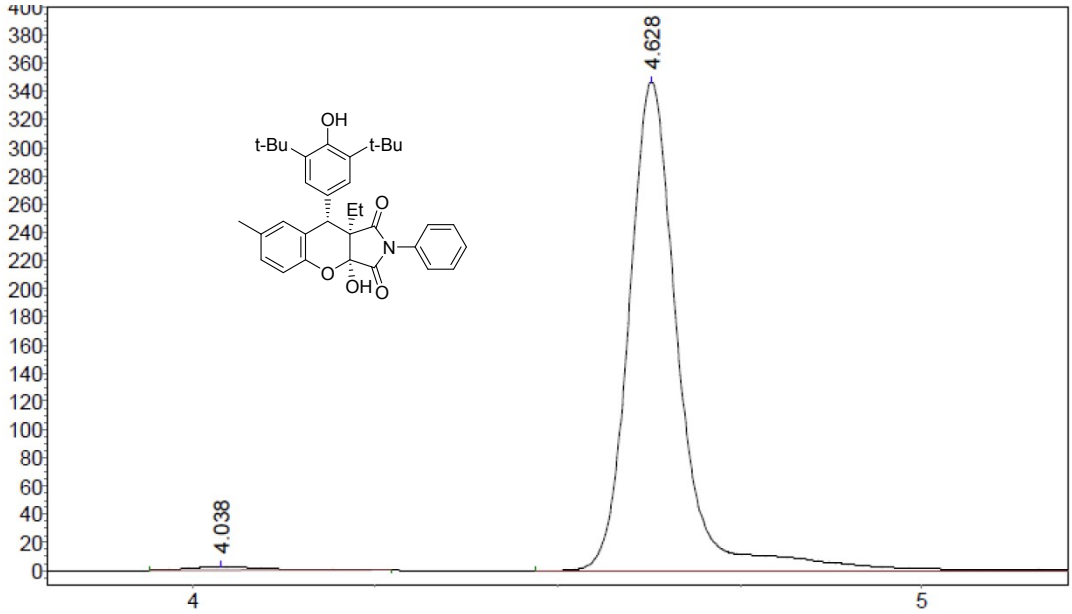


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.040	90008.516	732807.063	50.4983
2	4.627	154902.406	718344.125	49.5017

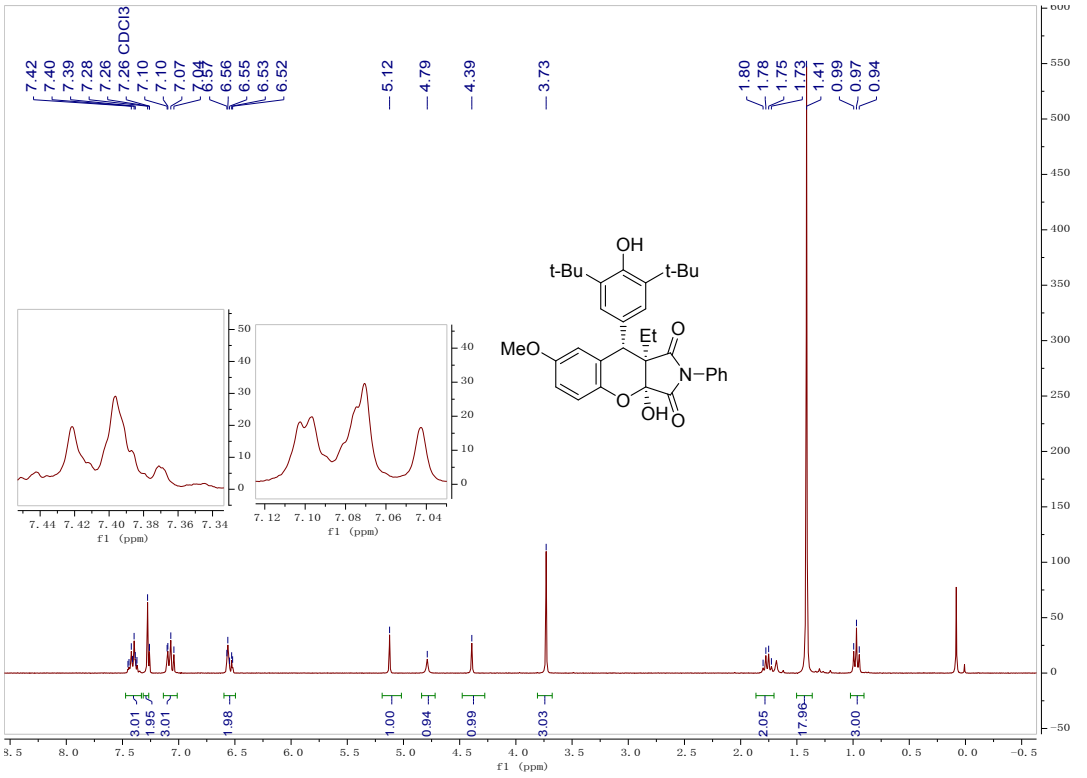


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.038	2421.763	19592.619	1.1799
2	4.628	346458.219	1640975.500	98.8201

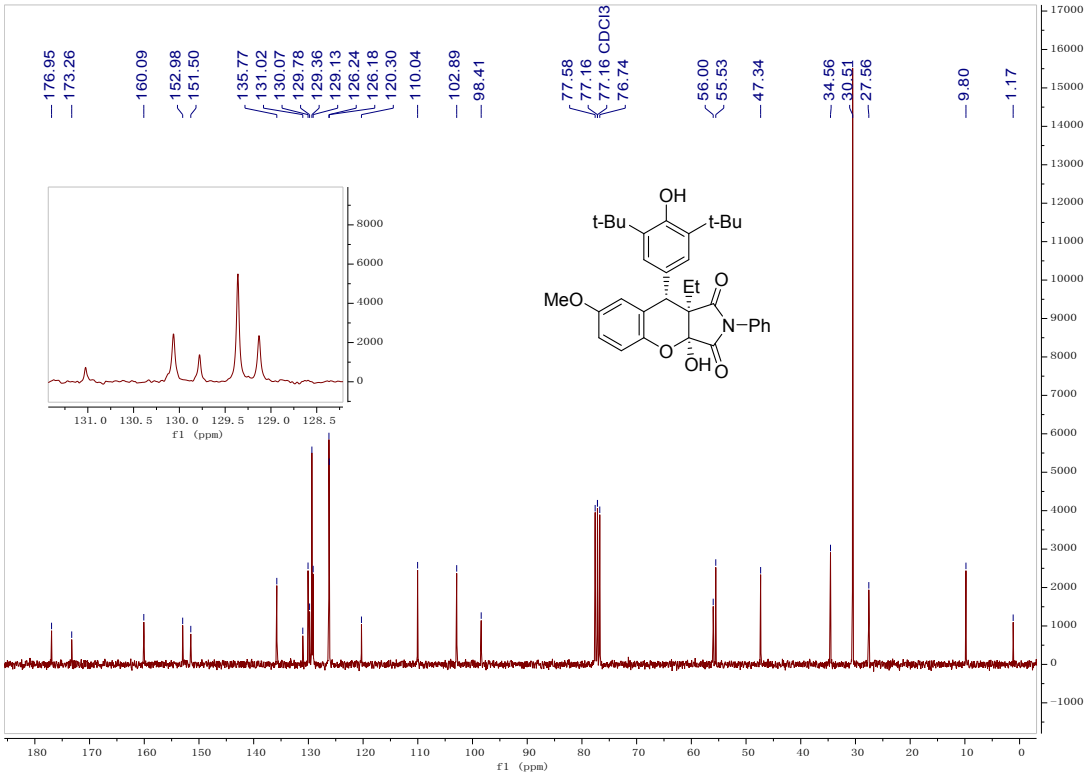
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.0 min, t_{major} = 4.6 min.

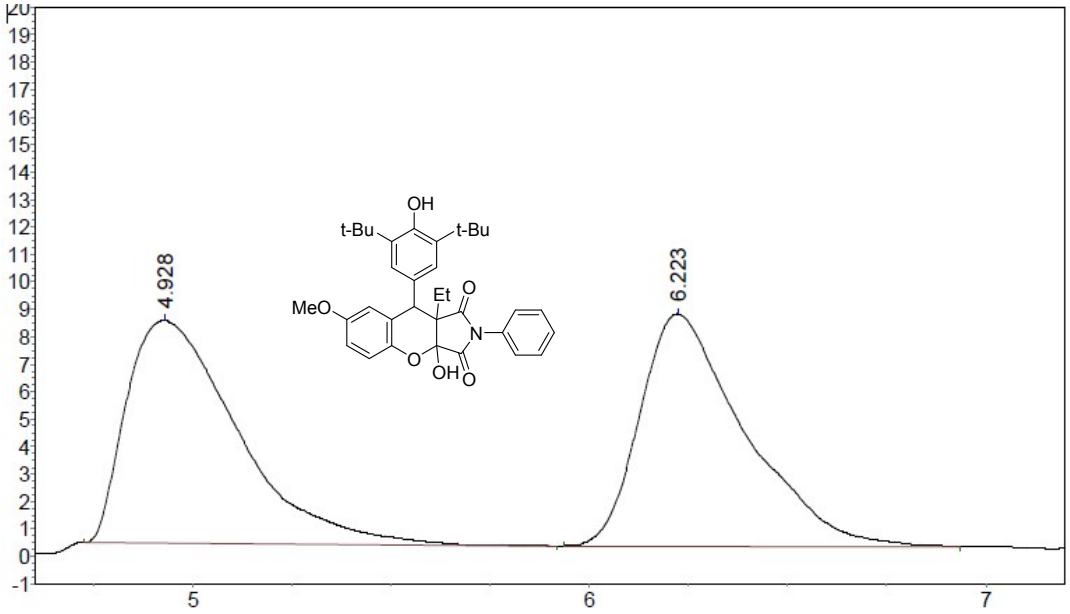
3ac

^{1}H NMR (300 MHz, $CDCl_3$)

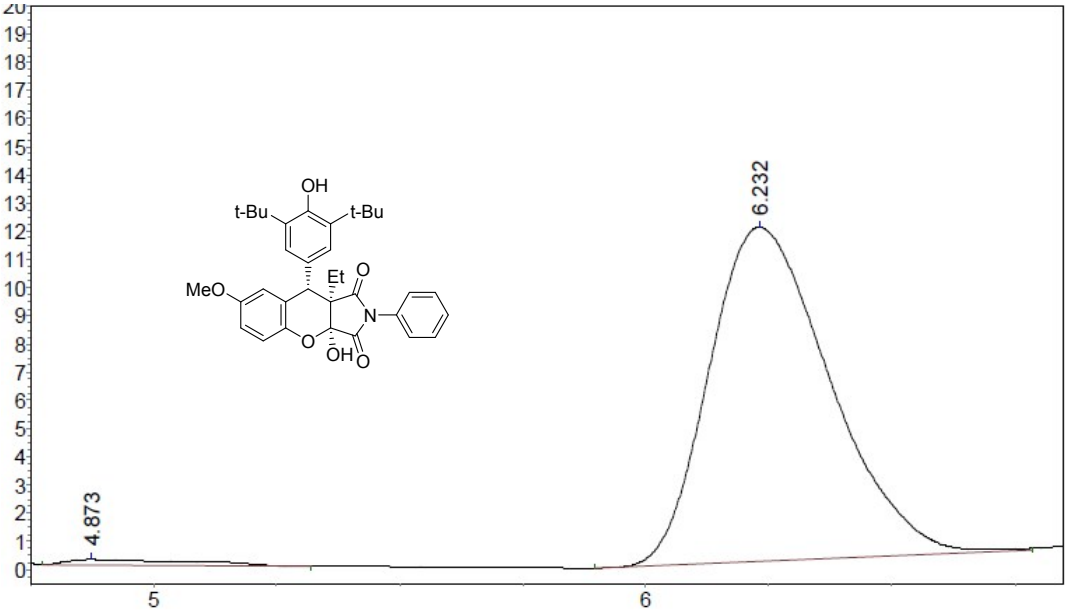


^{13}C NMR (75 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.928	29445.686	560574.875	49.1577
2	6.213	31863.857	579785.750	50.8423

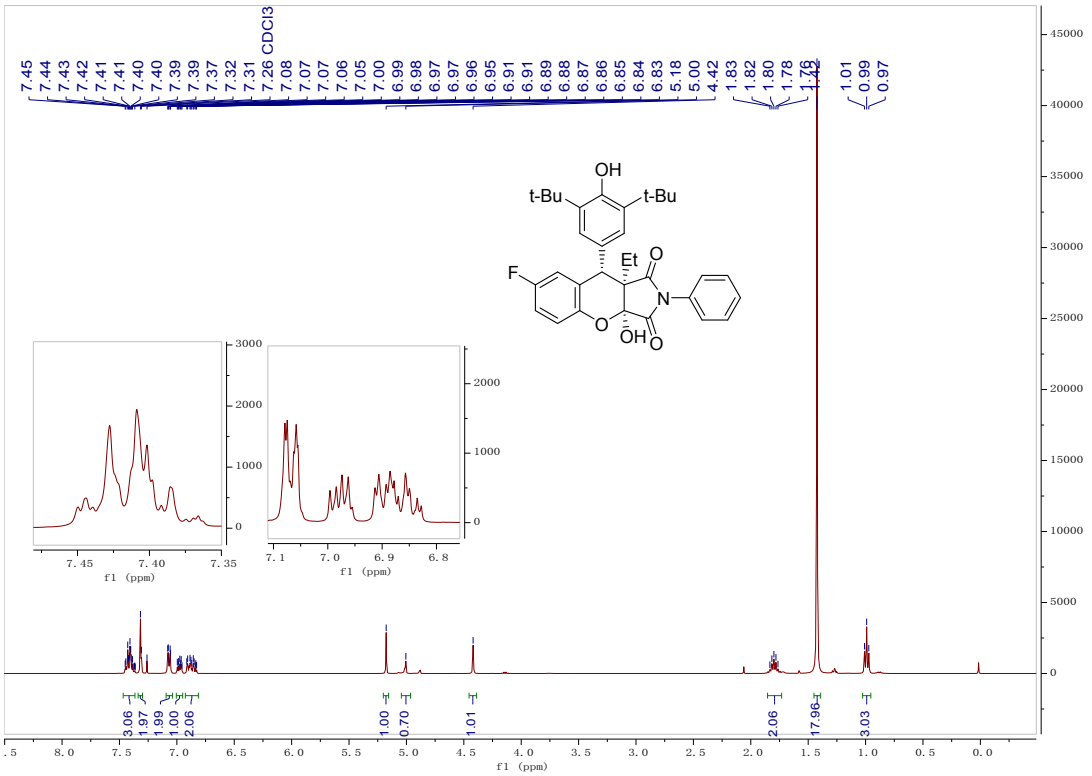


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.023	478.230	5853.676	2.1003
2	6.277	16394.590	272849.406	97.8997

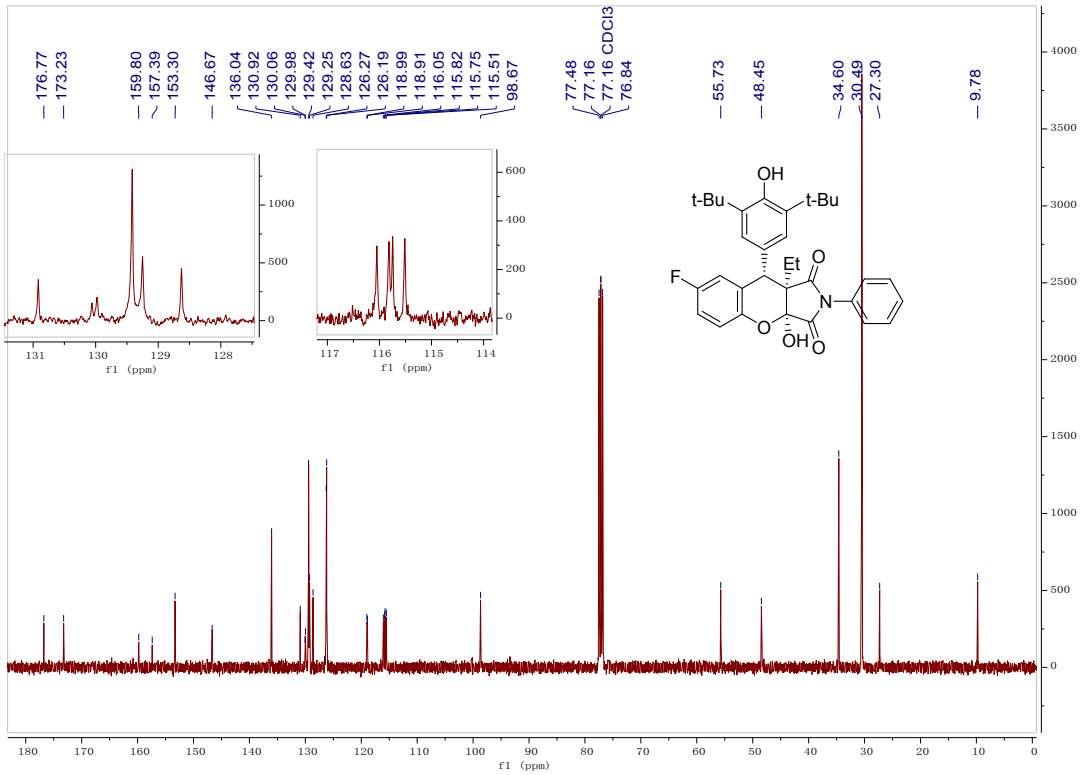
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 5.0 min, t_{major} = 6.2 min.

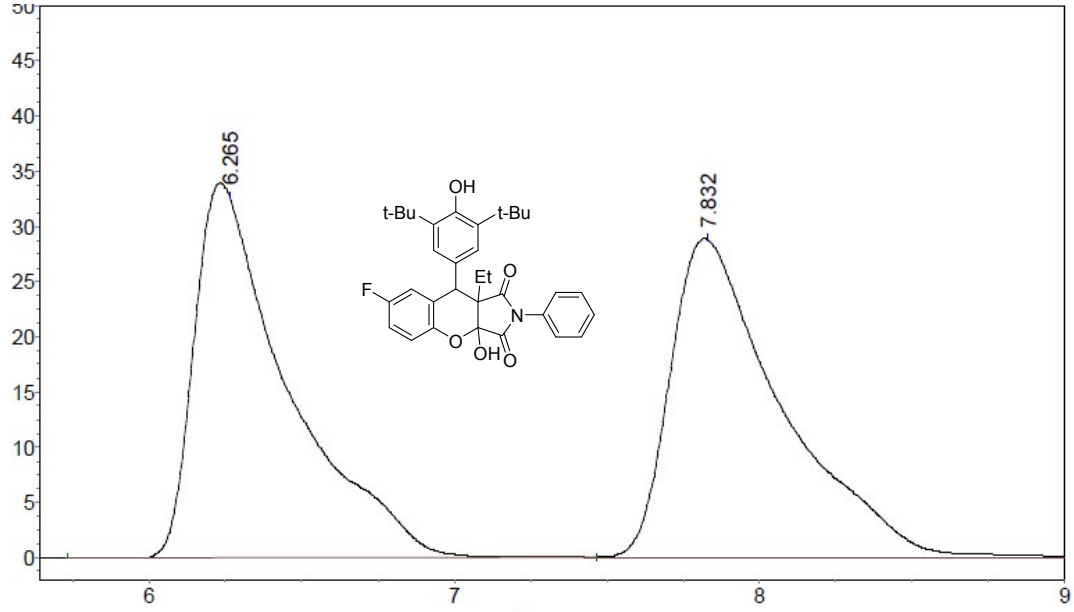
3ad

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

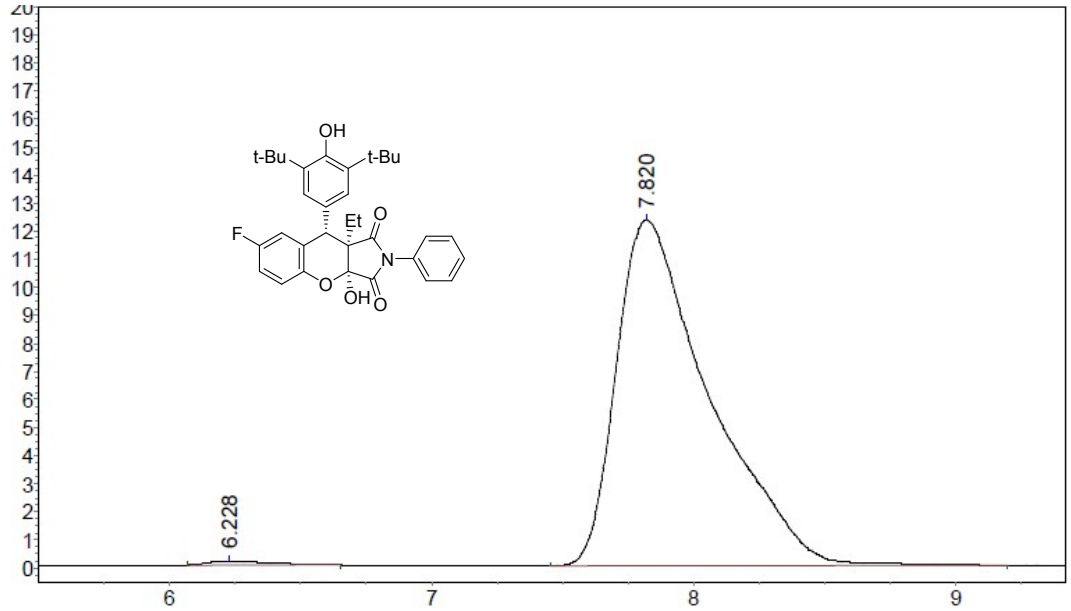


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	6.265	33449.070	721754.063	49.7736
2	7.832	28758.146	728320.375	50.2264

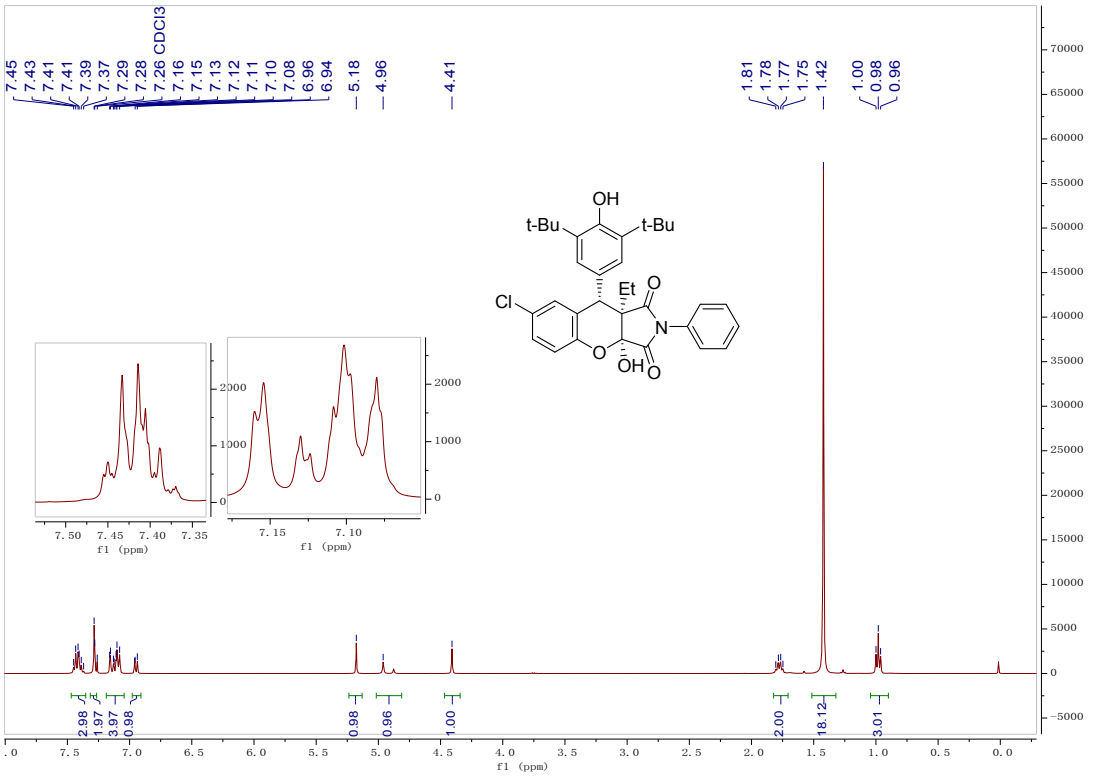


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	6.228	144.565	2425.017	0.7922
2	7.820	12314.445	303672.500	99.2078

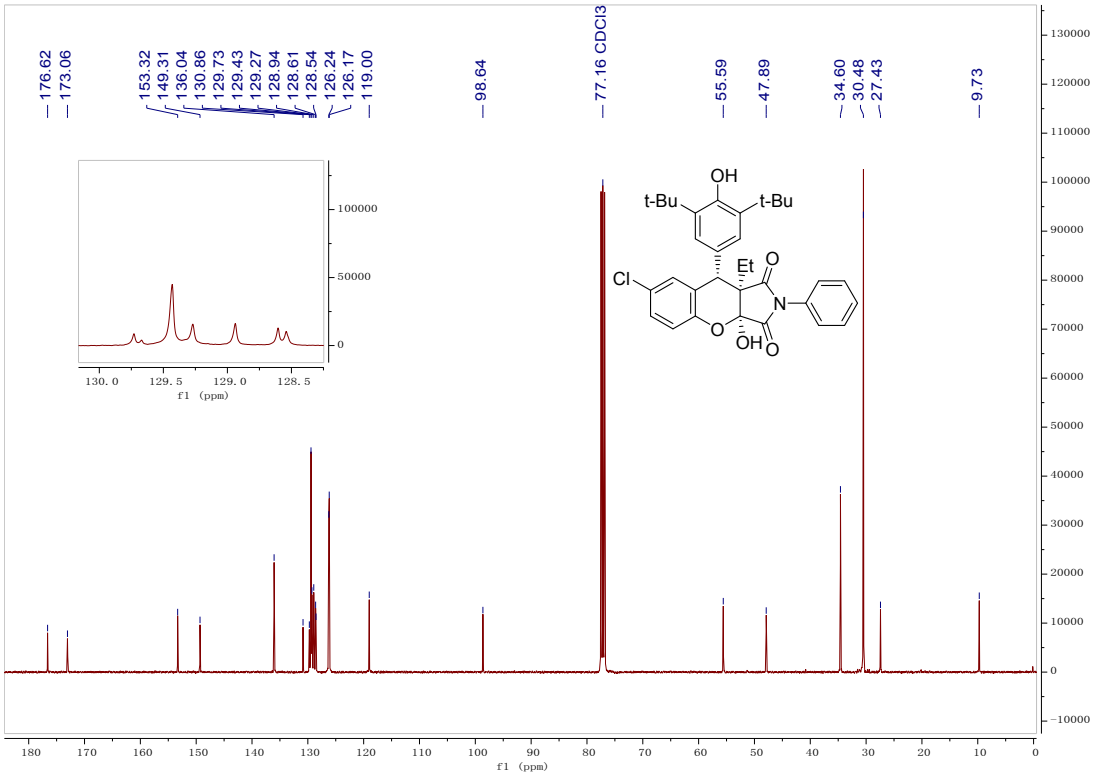
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 6.2 min, t_{major} = 7.8 min.

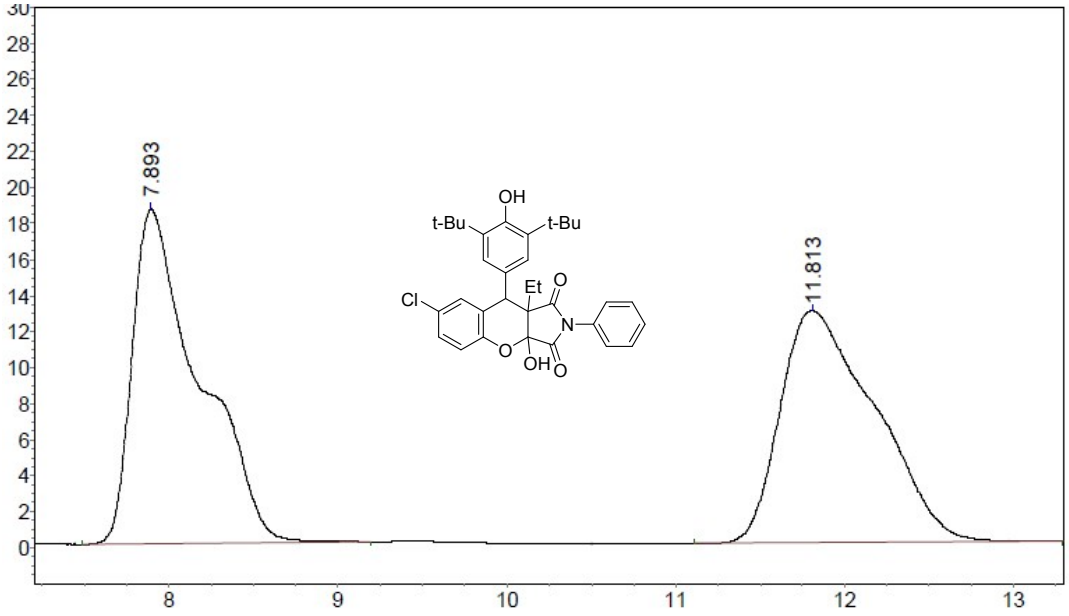
3ae

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

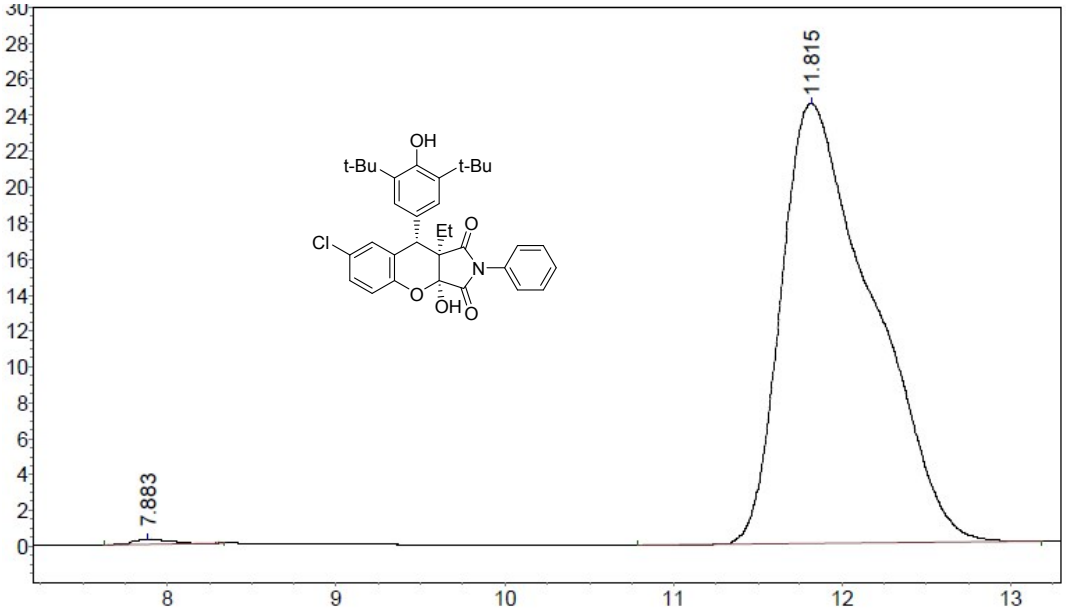


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	7.893	18528.346	507810.906	50.2346
2	11.813	12907.395	503068.313	49.7654

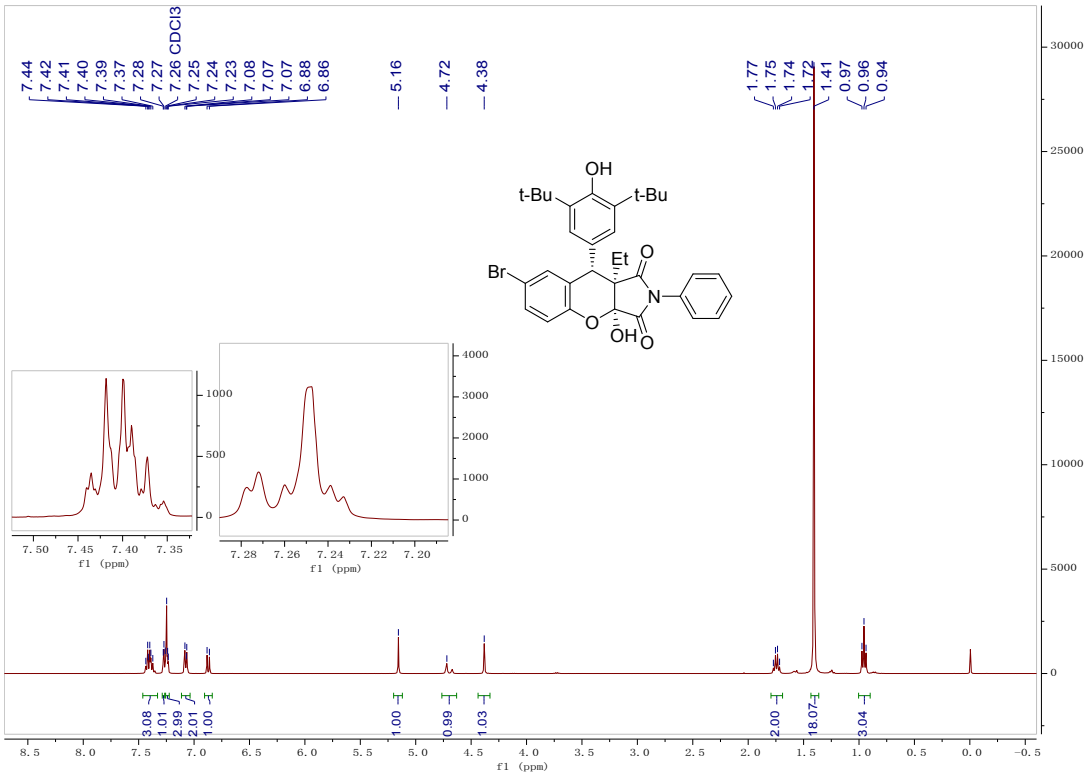


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	7.883	244.672	4201.689	0.4553
2	11.815	24495.338	918732.875	99.5447

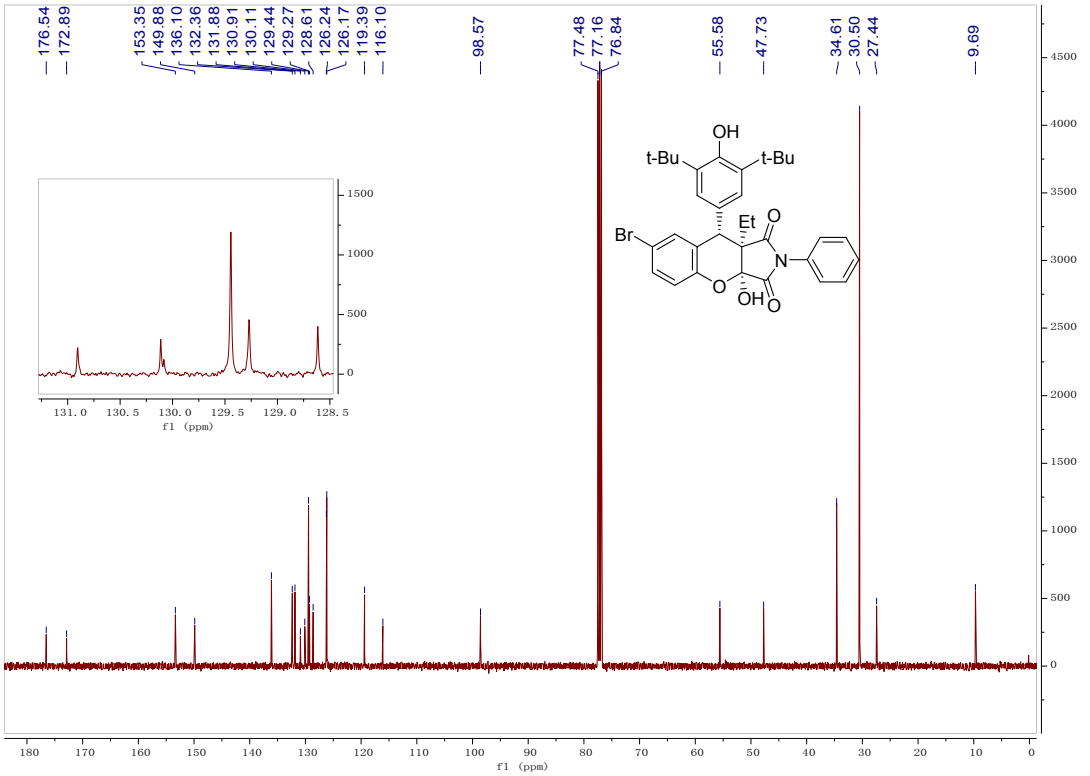
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 97/3, flow rate 1mL/min, λ = 254 nm, t_{minor} = 7.8 min, t_{major} = 11.8 min.

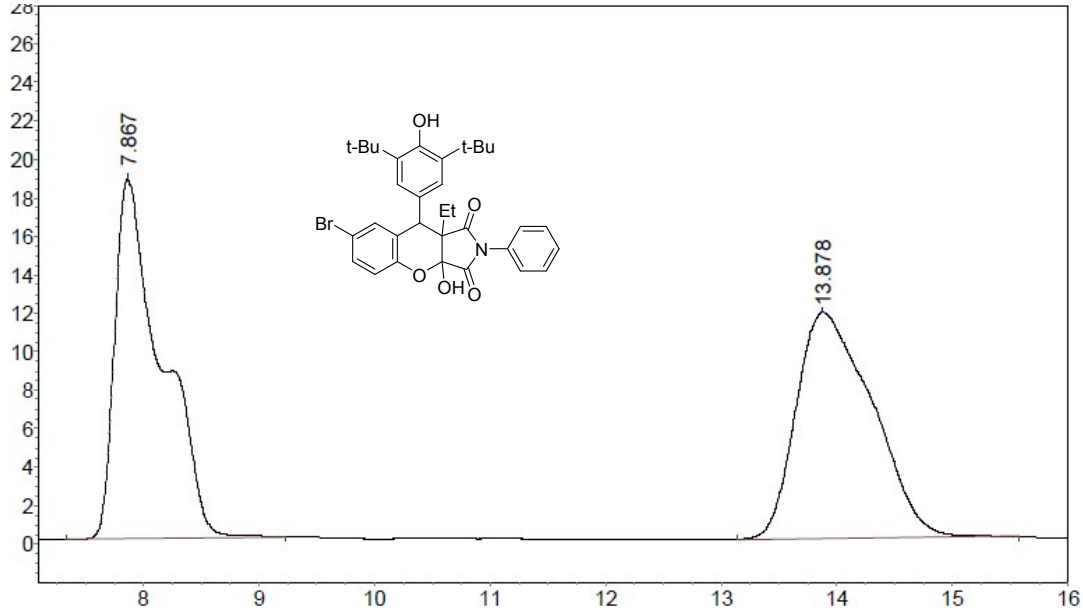
3af

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

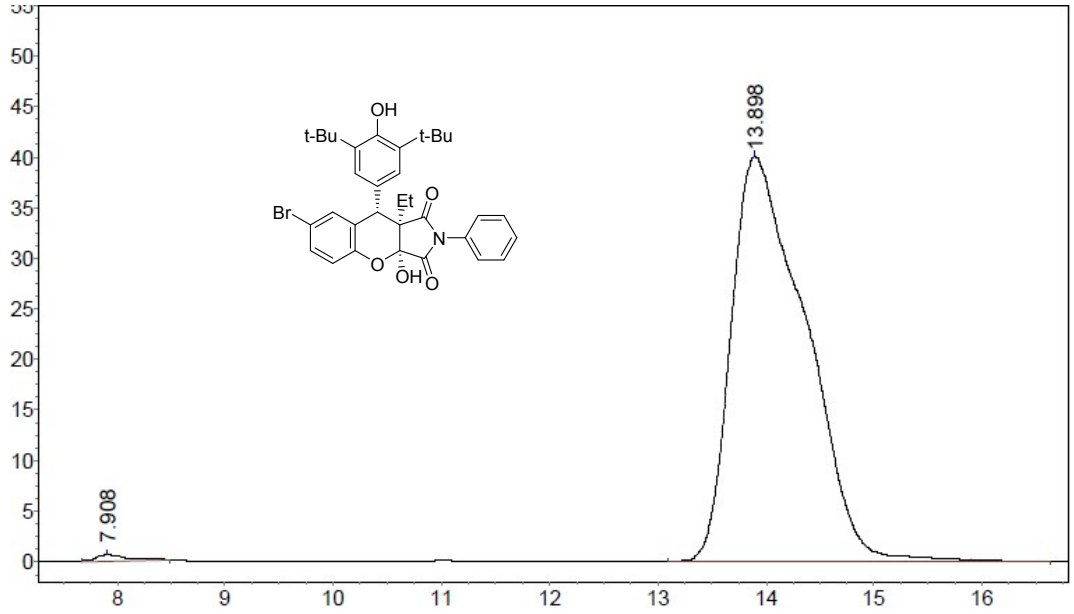


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	7.867	18719.461	524734.313	49.3579
2	13.878	11756.363	538386.000	50.6421

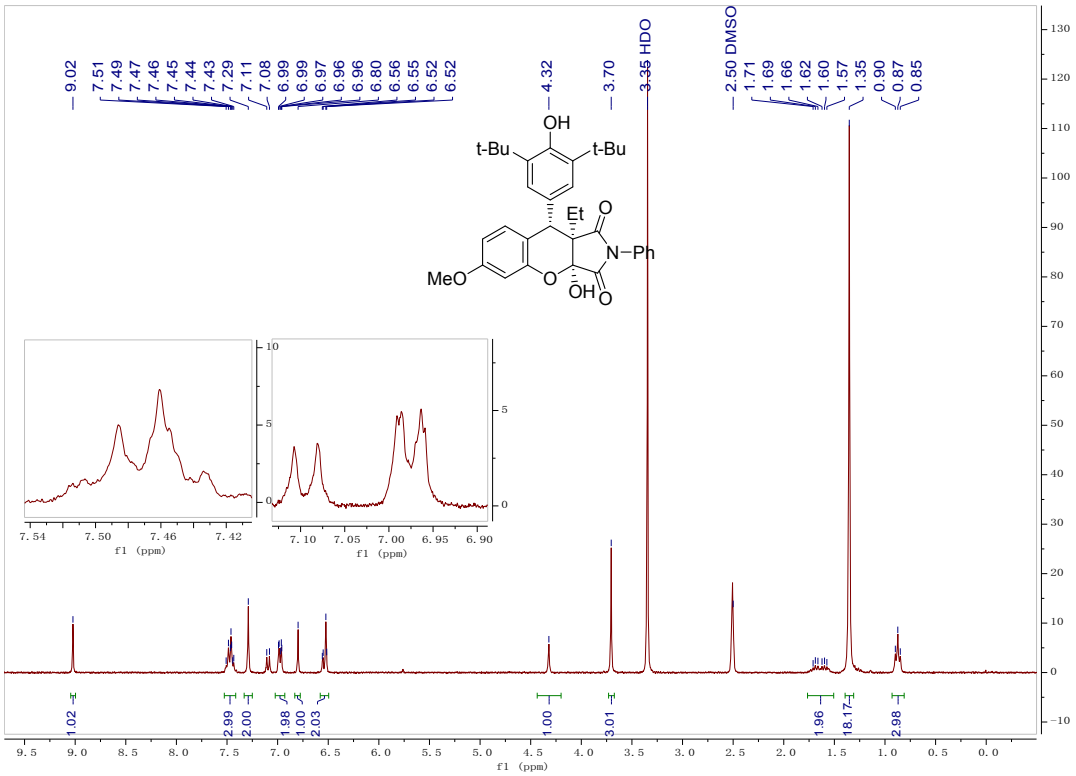


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	7.908	567.380	12450.849	0.6480
2	13.898	39992.488	1909039.000	99.3520

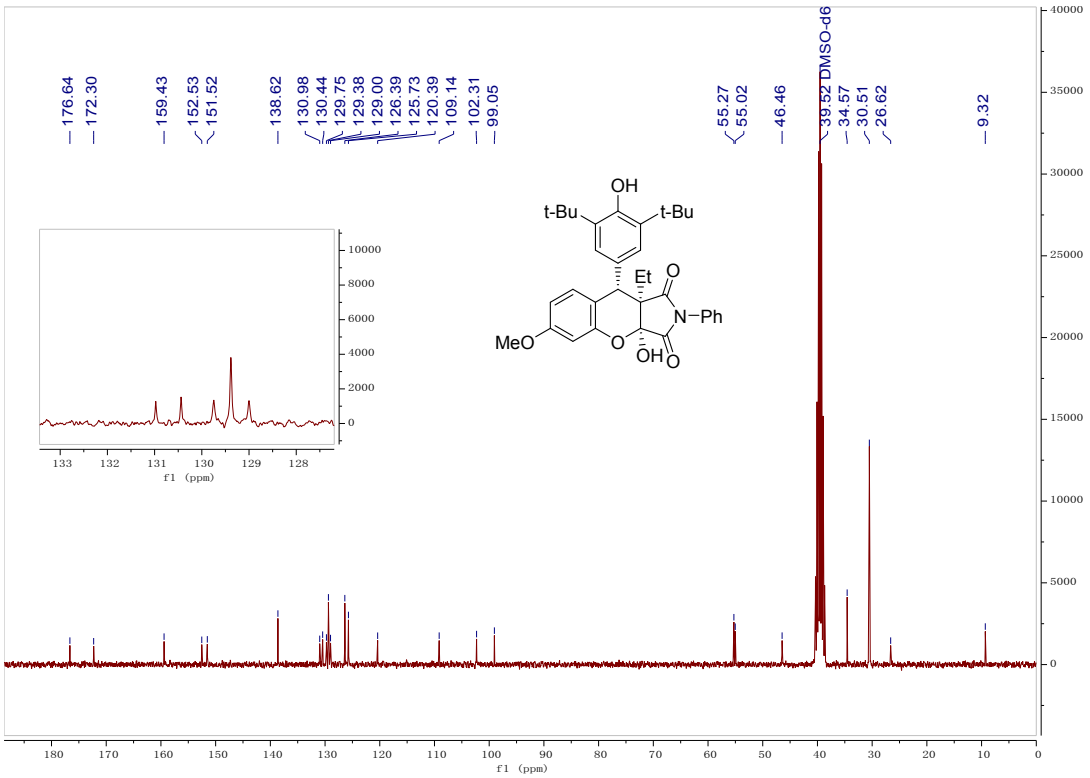
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 97/3, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 7.9 min, t_{major} = 13.8 min.

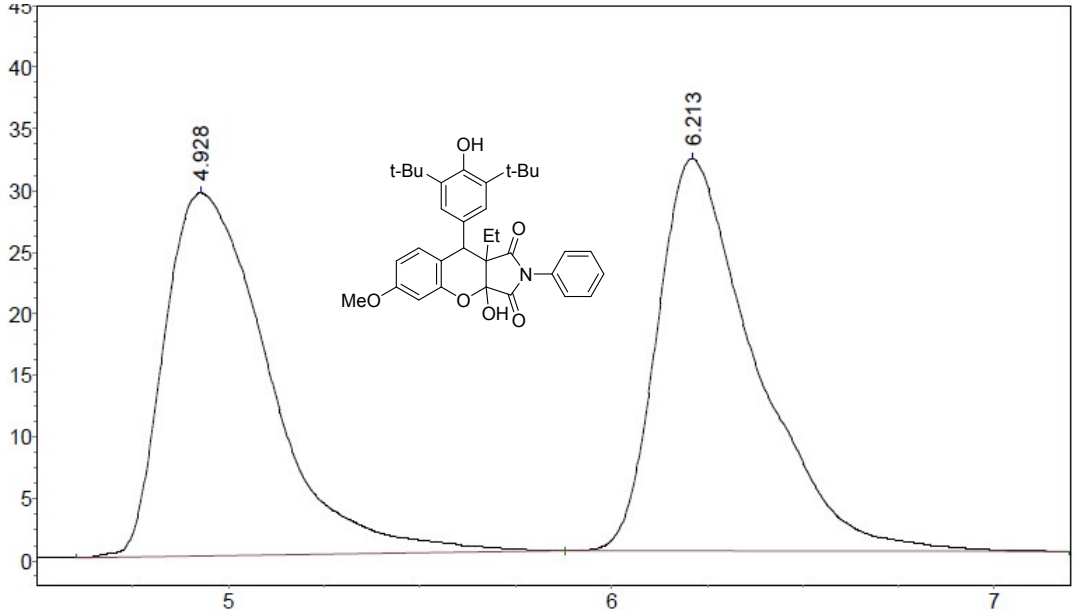
3ag

^{1}H NMR (300 MHz, DMSO-d_6)

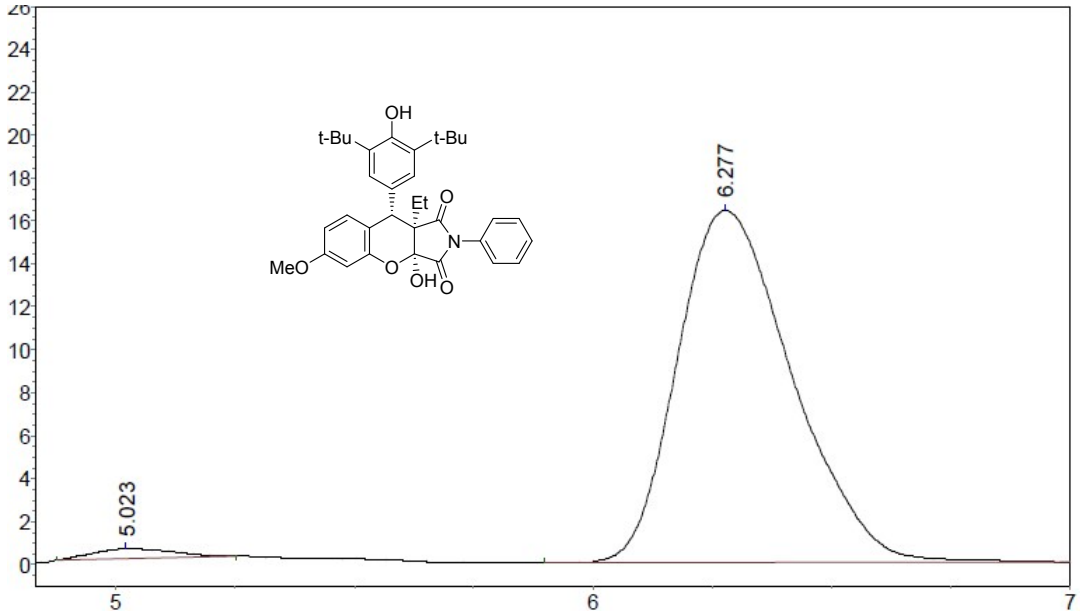


^{13}C NMR (300 MHz, DMSO-d6)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.928	29445.686	560574.875	49.1577
2	6.213	31863.857	579785.750	50.8423

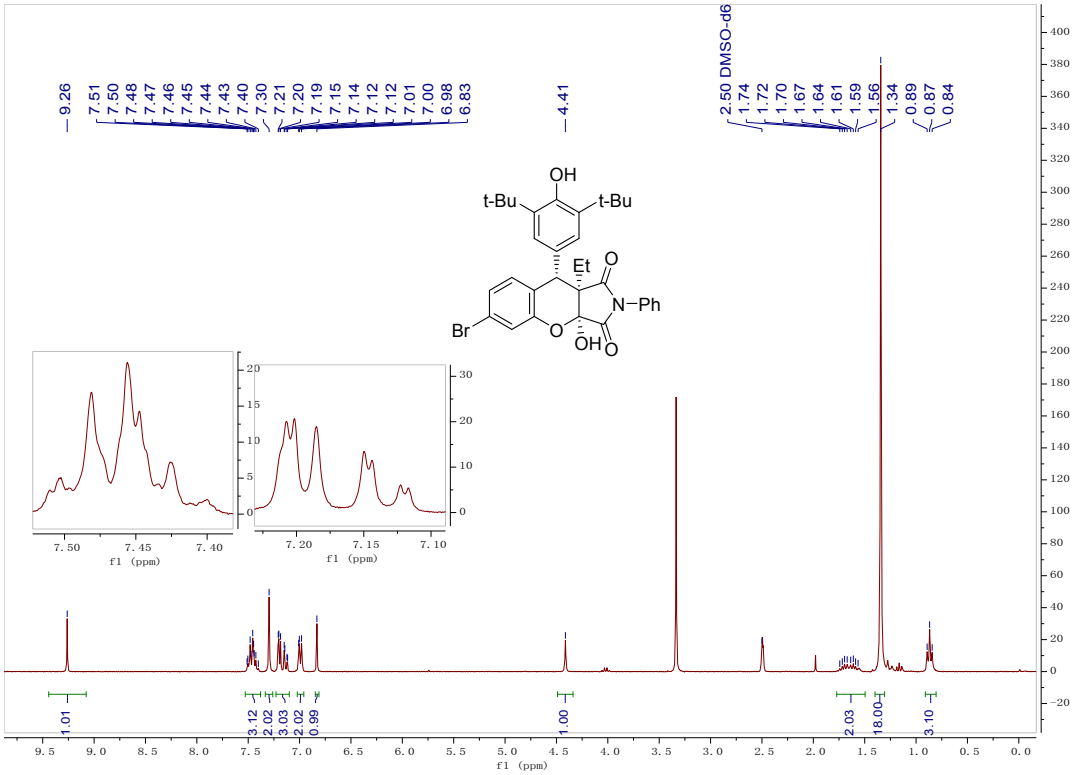


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.873	217.222	3996.050	1.9434
2	6.232	11847.739	201622.922	98.0566

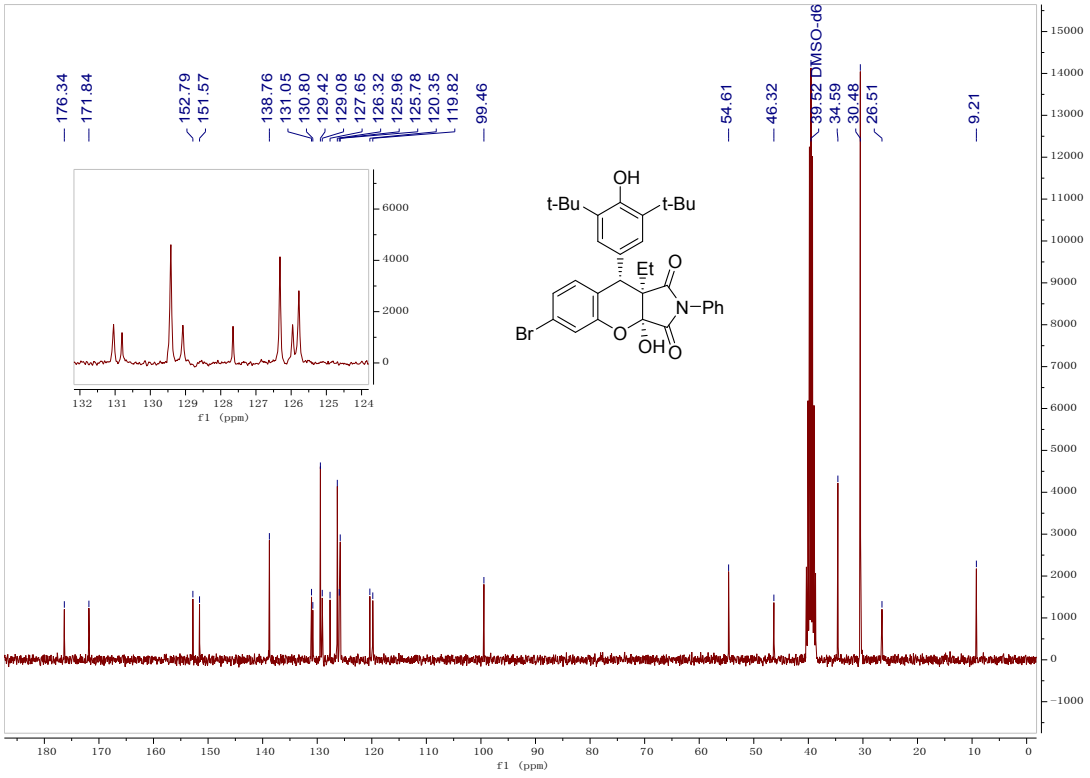
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.8 min, t_{major} = 6.2 min.

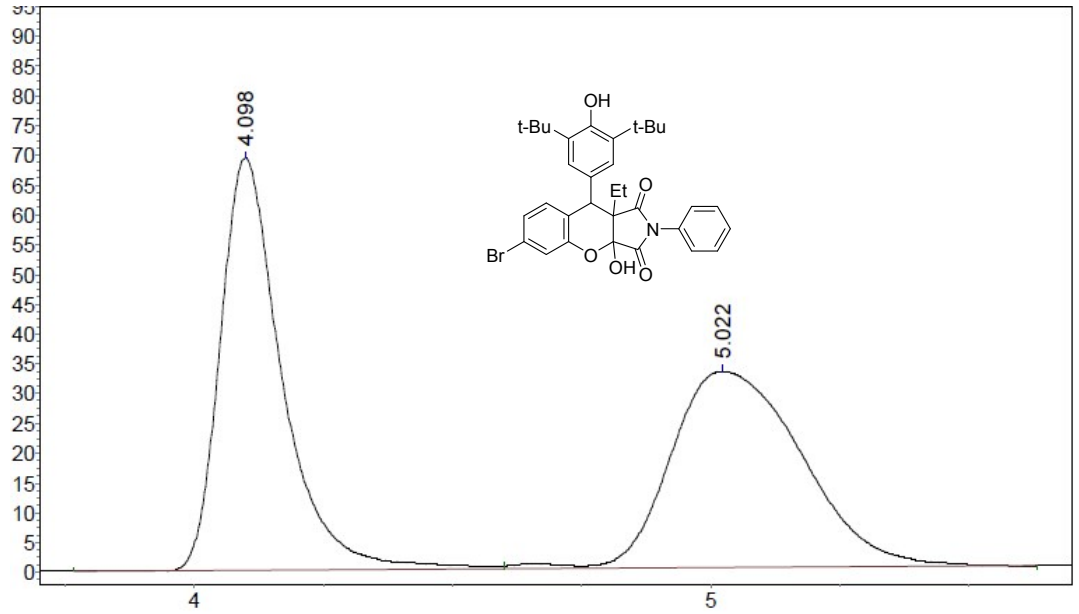
3ah

^{1}H NMR (300 MHz, DMSO-d^{6})

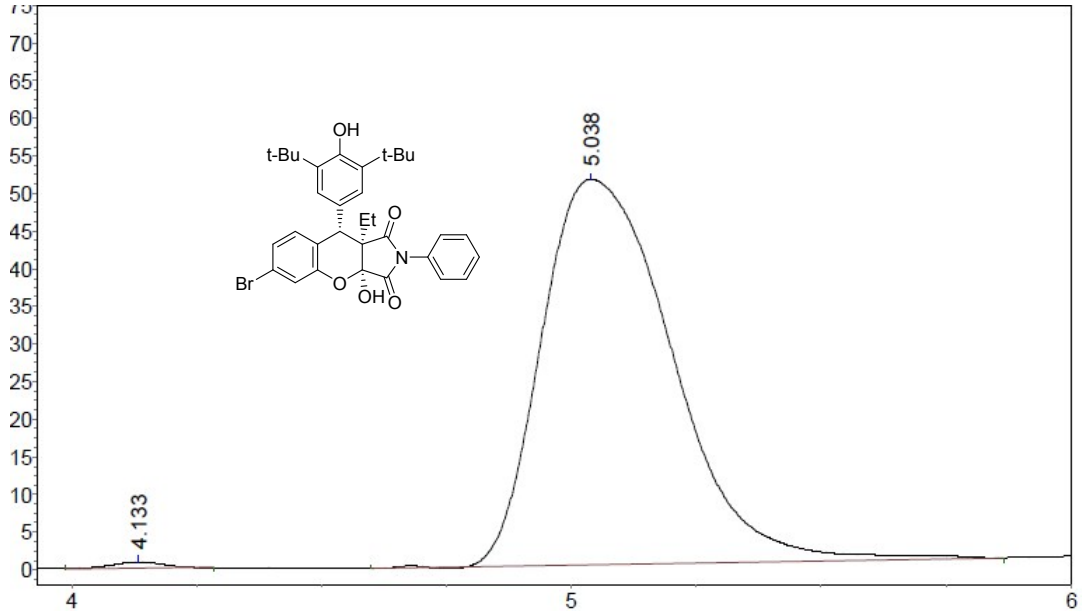


^{13}C NMR (75 MHz, DMSO-d^{6})





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.098	69189.523	575132.500	49.6879
2	5.022	32987.391	582357.625	50.3121

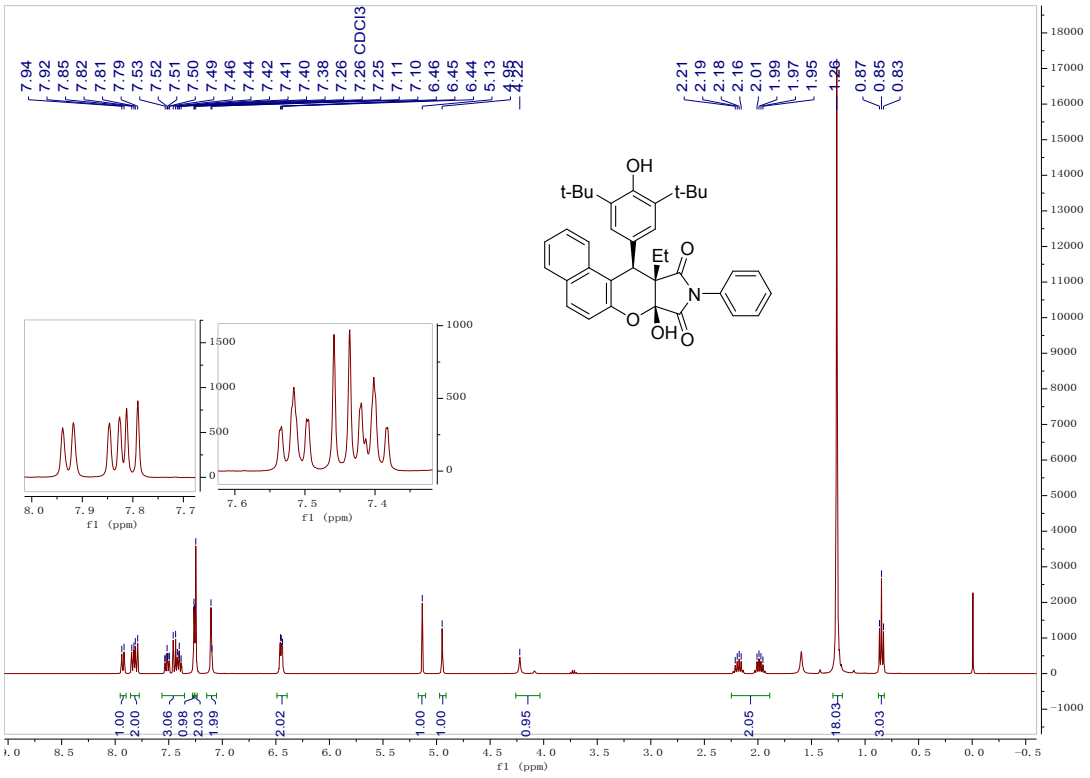


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.133	844.225	6127.569	0.6561
2	5.038	51273.383	927796.188	99.3439

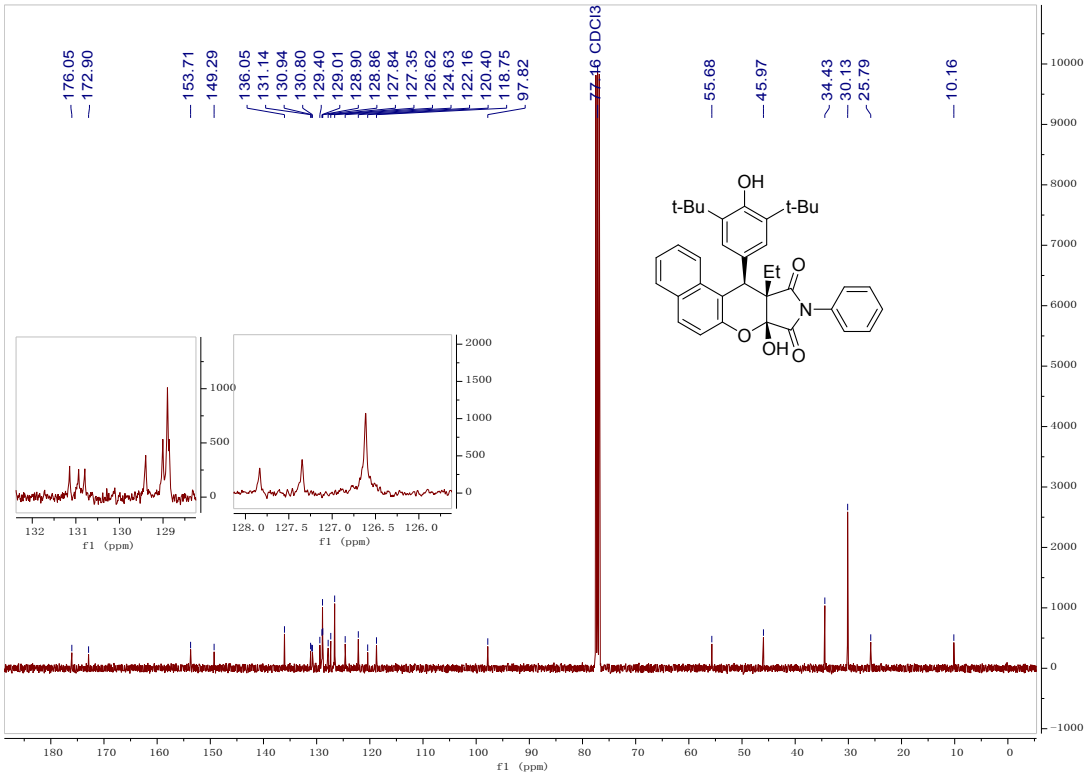
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.1min, t_{major} =5.0 min.

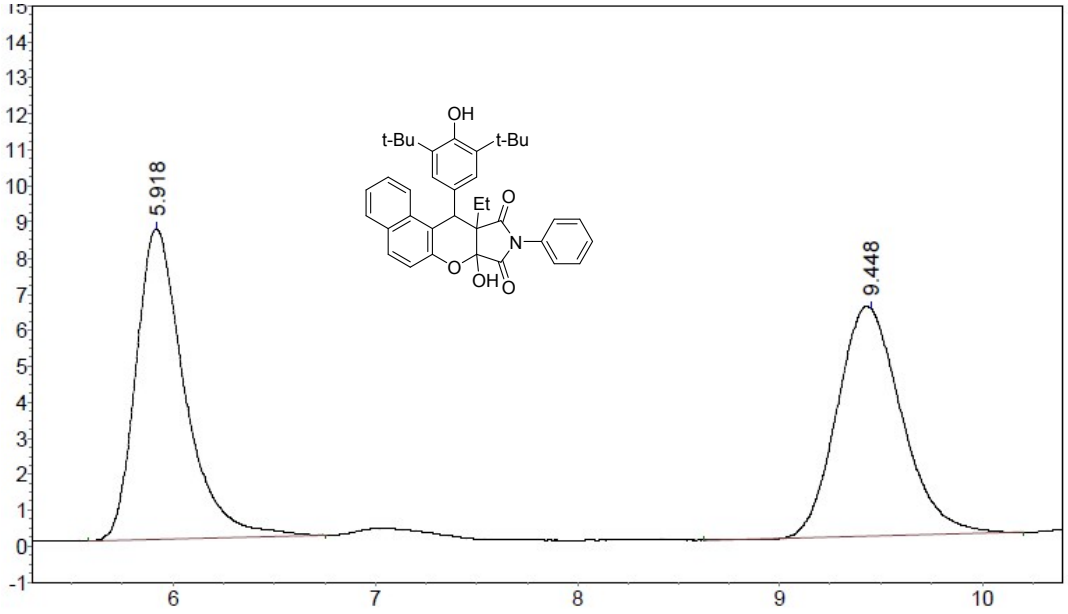
3ai

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

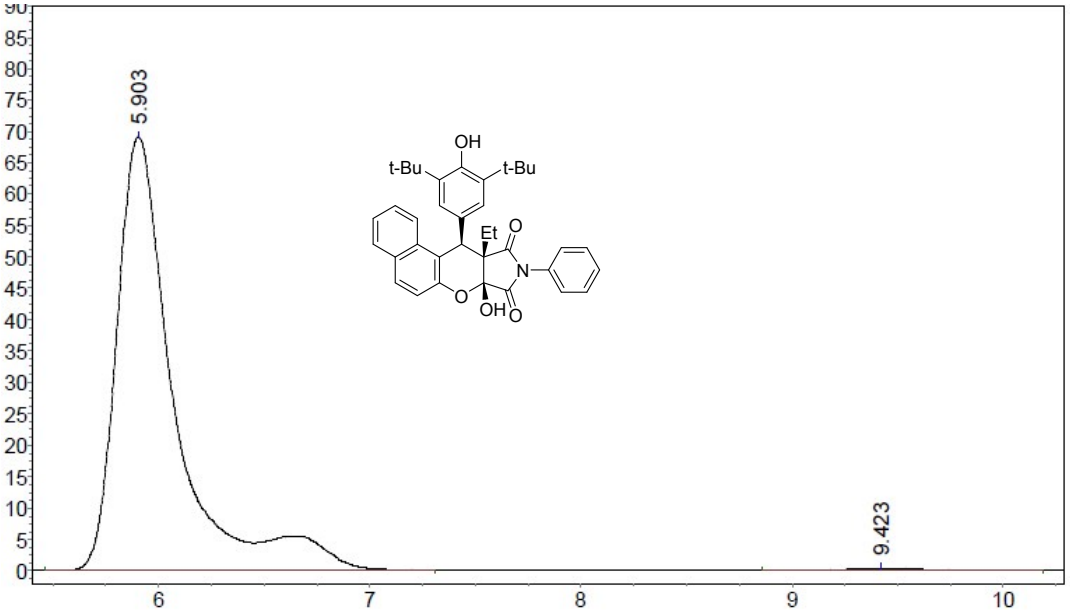


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.918	8656.133	149452.875	49.9659
2	9.448	6462.412	149656.609	50.0341

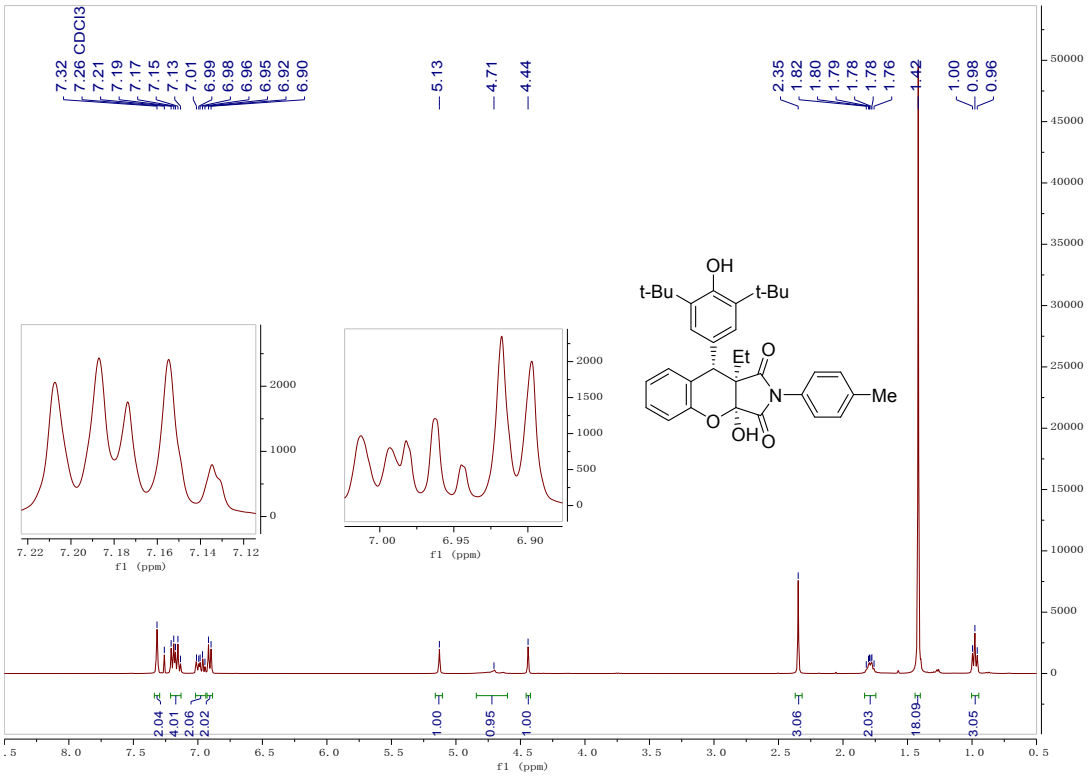


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.903	68987.672	1315557.375	99.3981
2	9.423	375.160	7965.976	0.6019

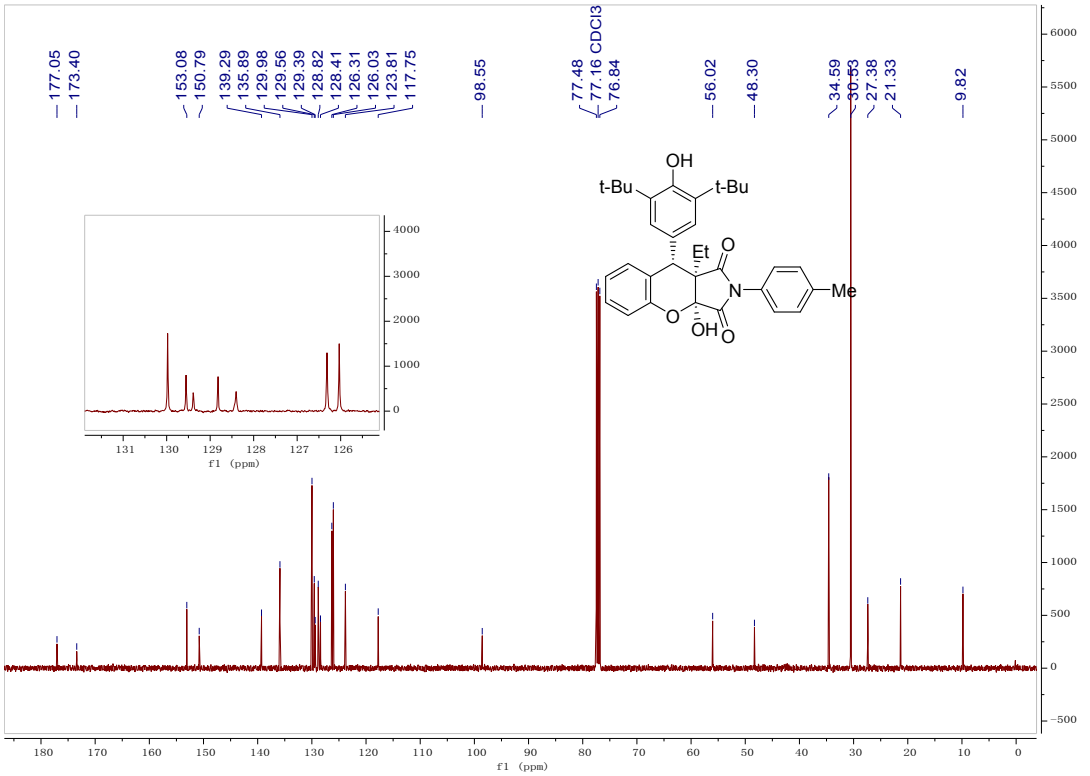
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 5.9 min, t_{major} =9.4 min.

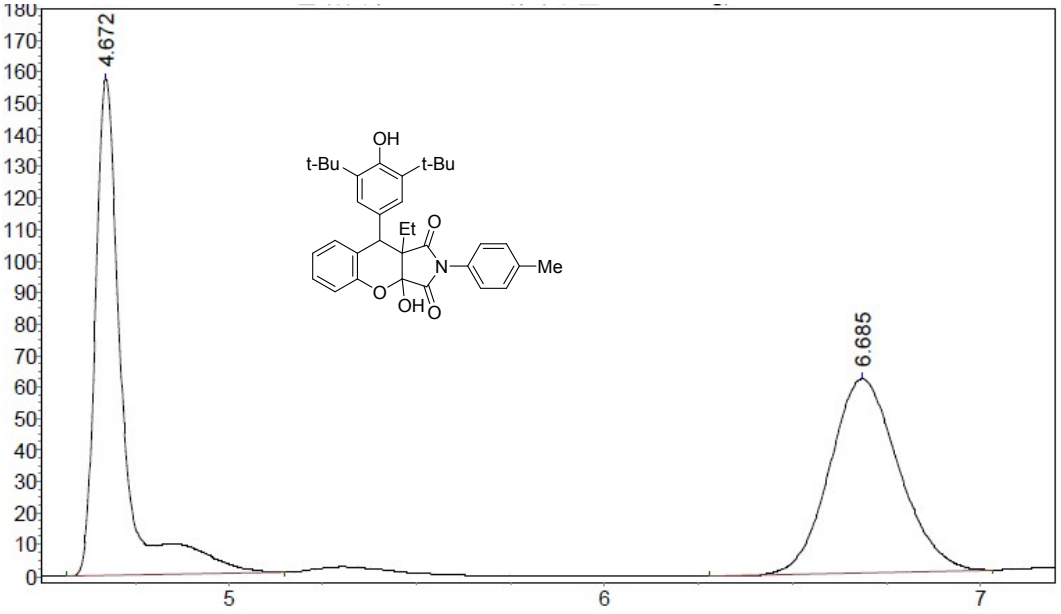
3ba

1H NMR (400 MHz, $CDCl_3$)

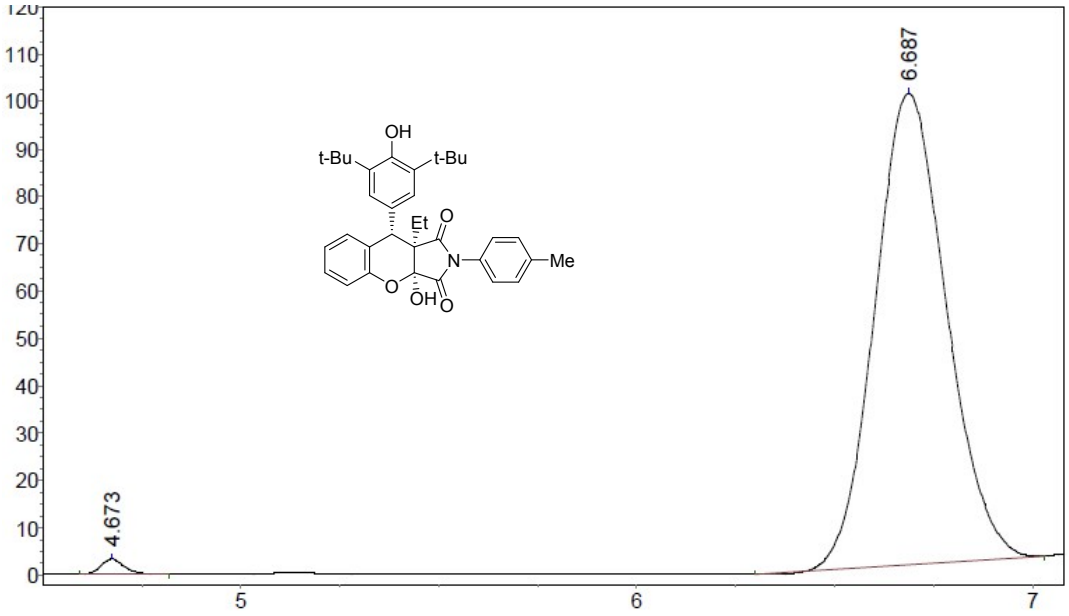


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.672	157236.531	783670.313	49.9779
2	6.685	61462.859	784362.000	50.0221

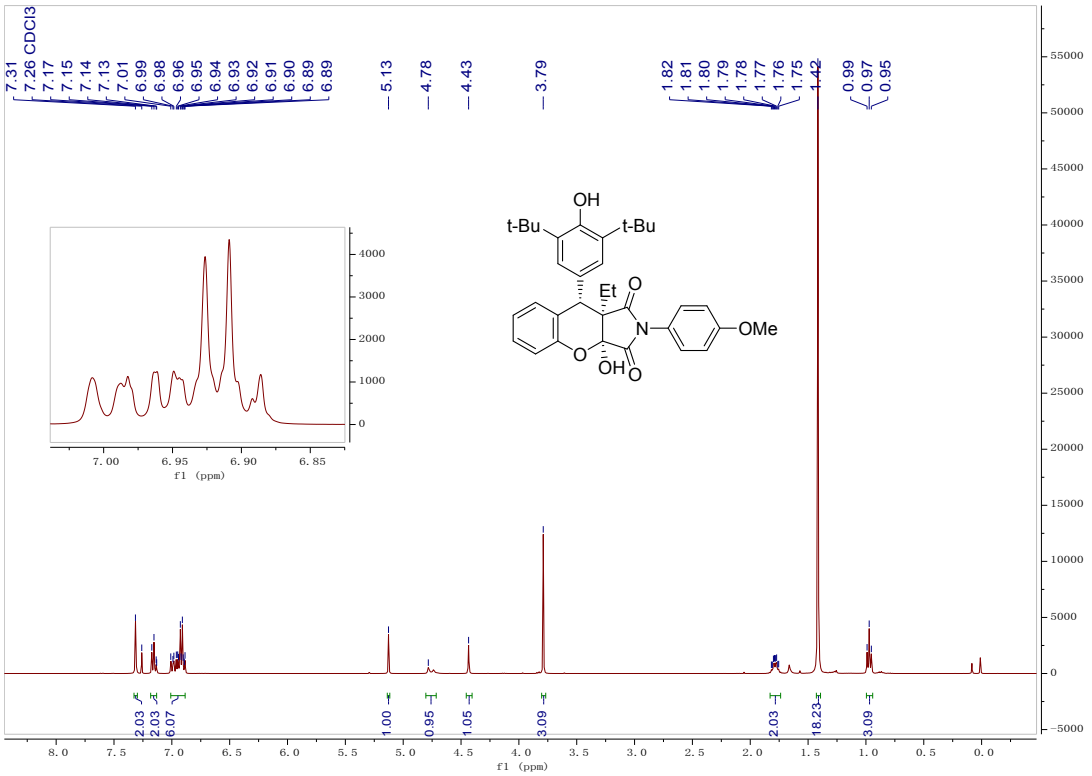


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.673	3252.177	13507.899	1.0705
2	6.687	99578.961	1248270.500	98.9295

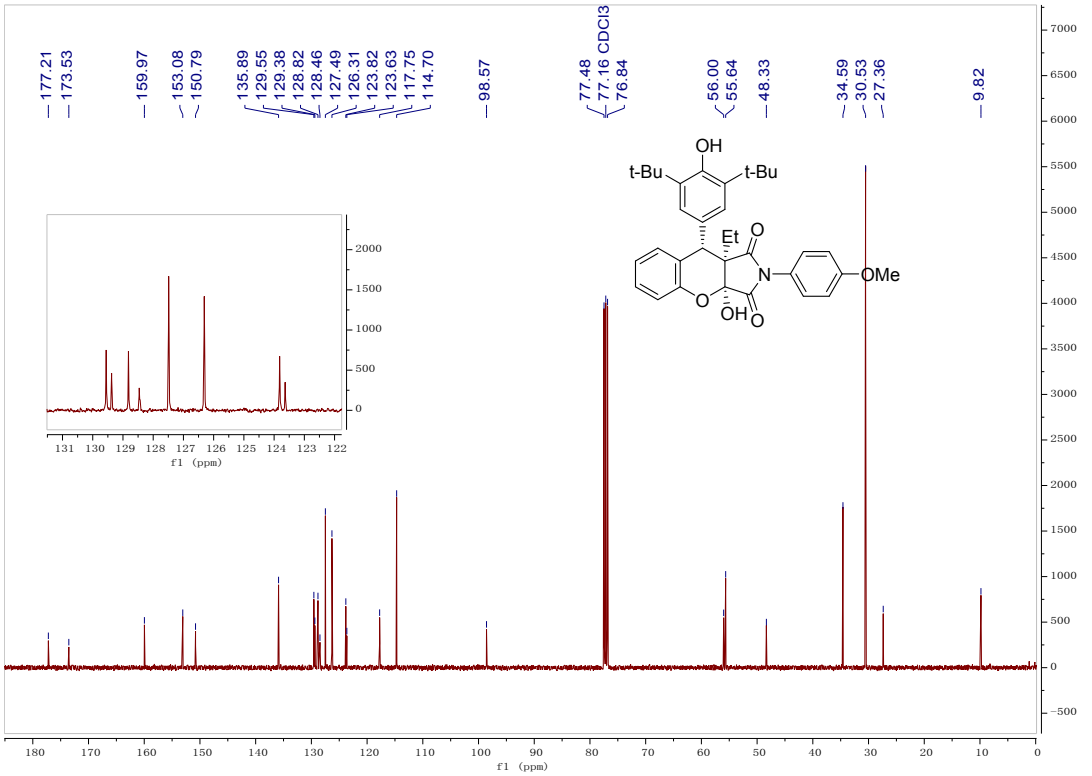
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.6 min, t_{major} = 6.6 min.

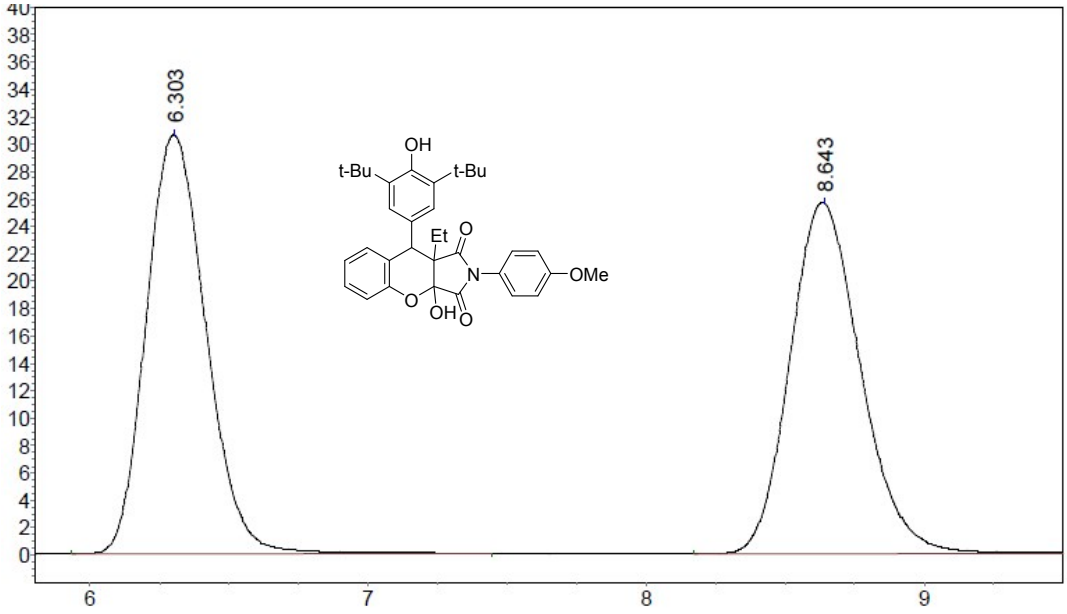
3ca

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

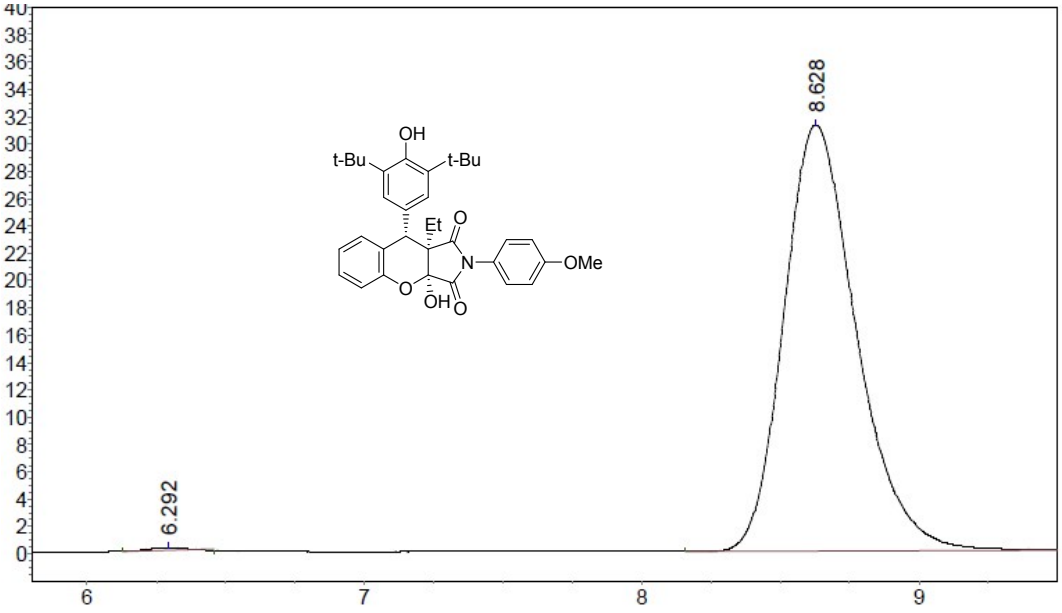


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	6.303	30586.385	457543.188	50.1206
2	8.643	25620.307	455341.000	49.8794

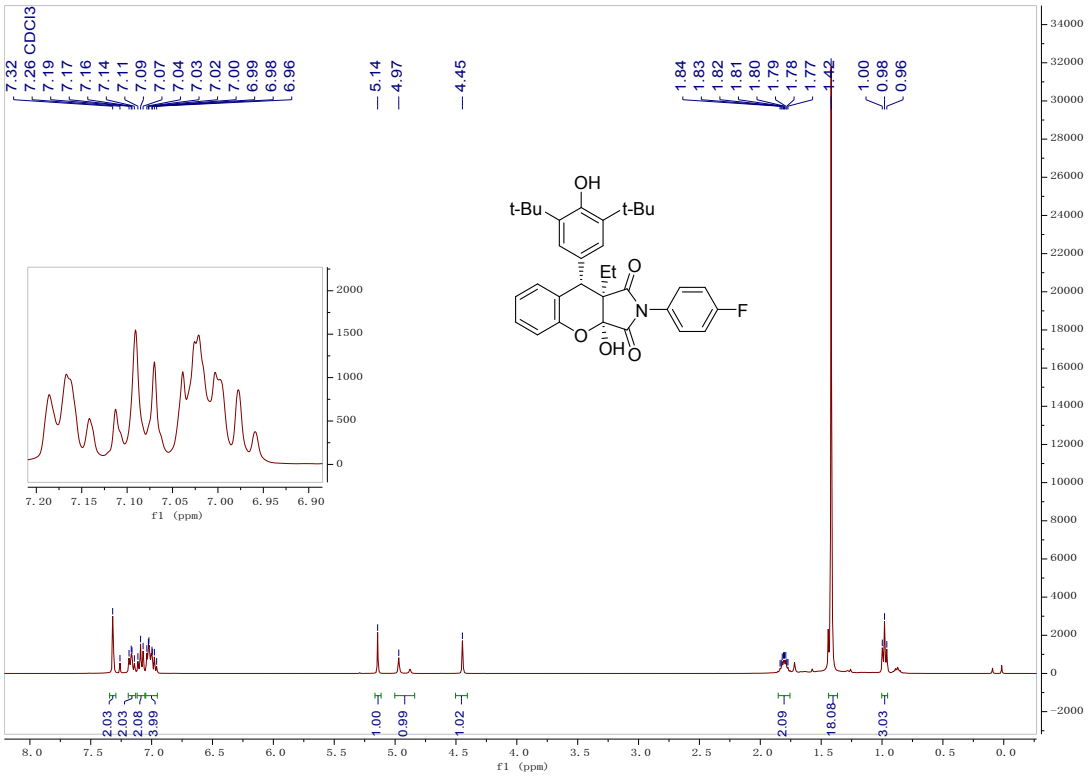


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	6.292	204.266	1822.251	0.3192
2	8.628	31176.430	569018.875	99.6808

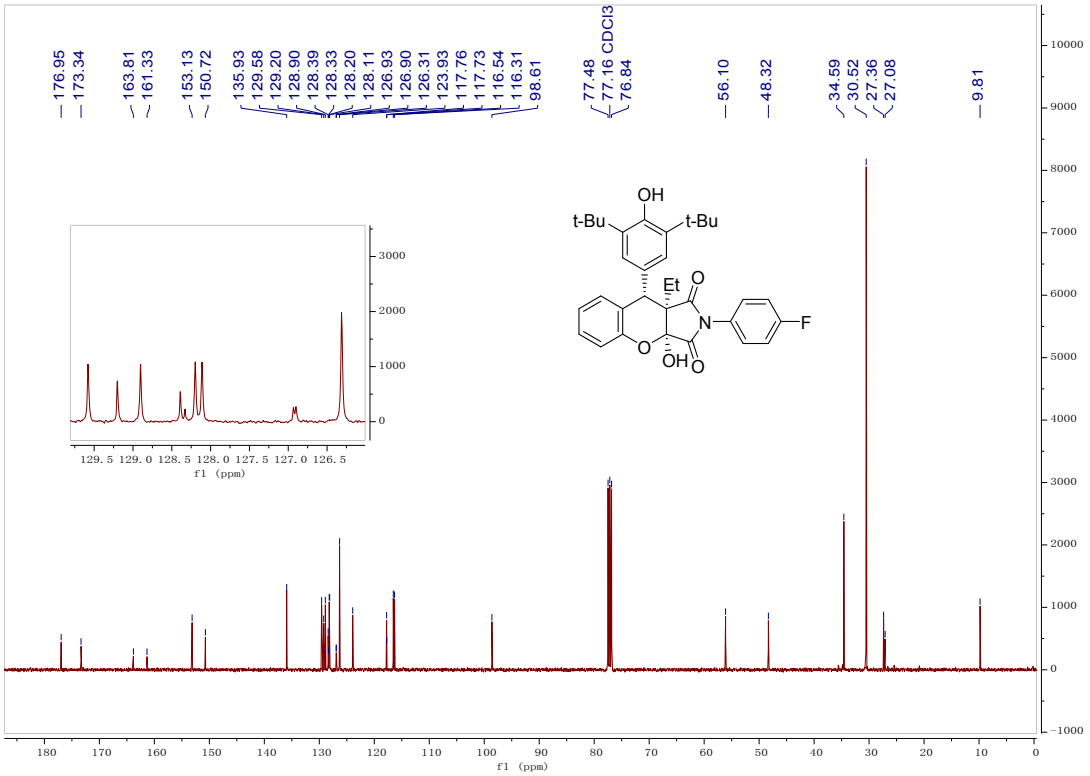
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 6.2 min, t_{major} = 8.6 min.

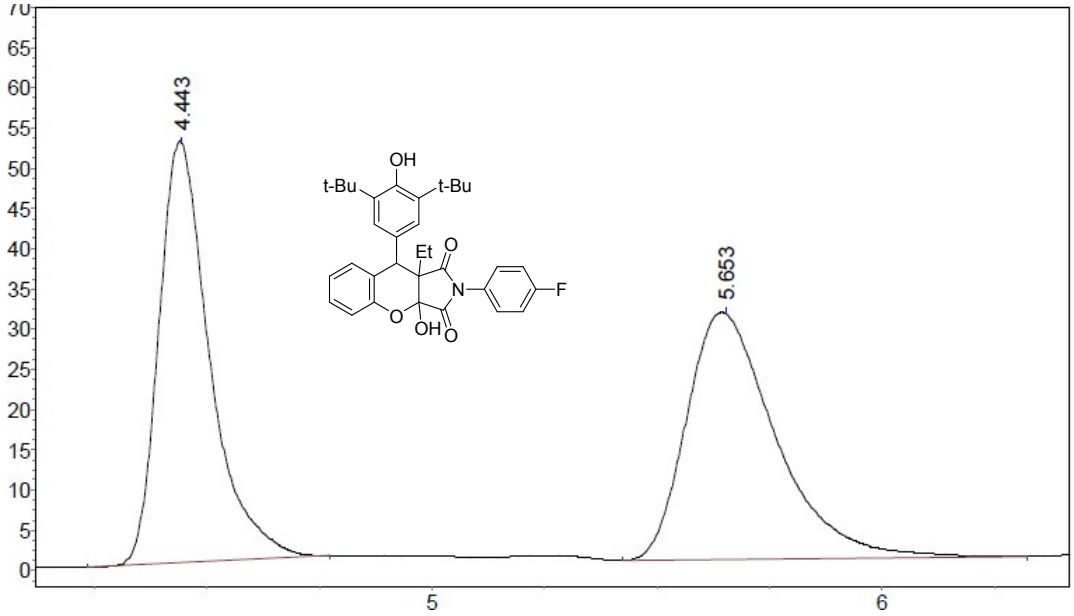
3da

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

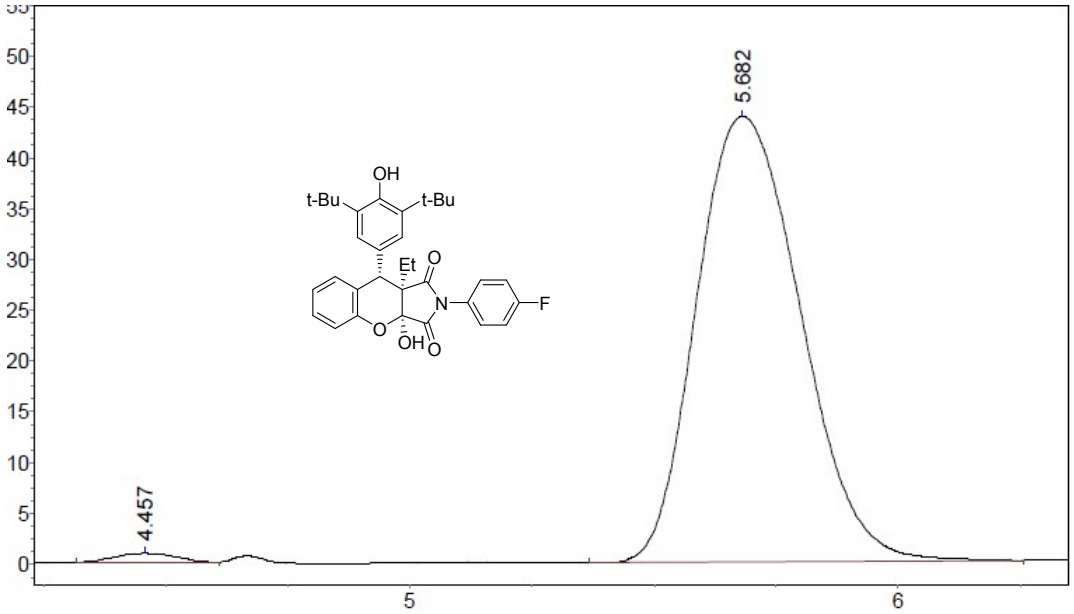


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.443	52447.285	418215.219	49.5461
2	5.653	30710.434	425878.594	50.4539

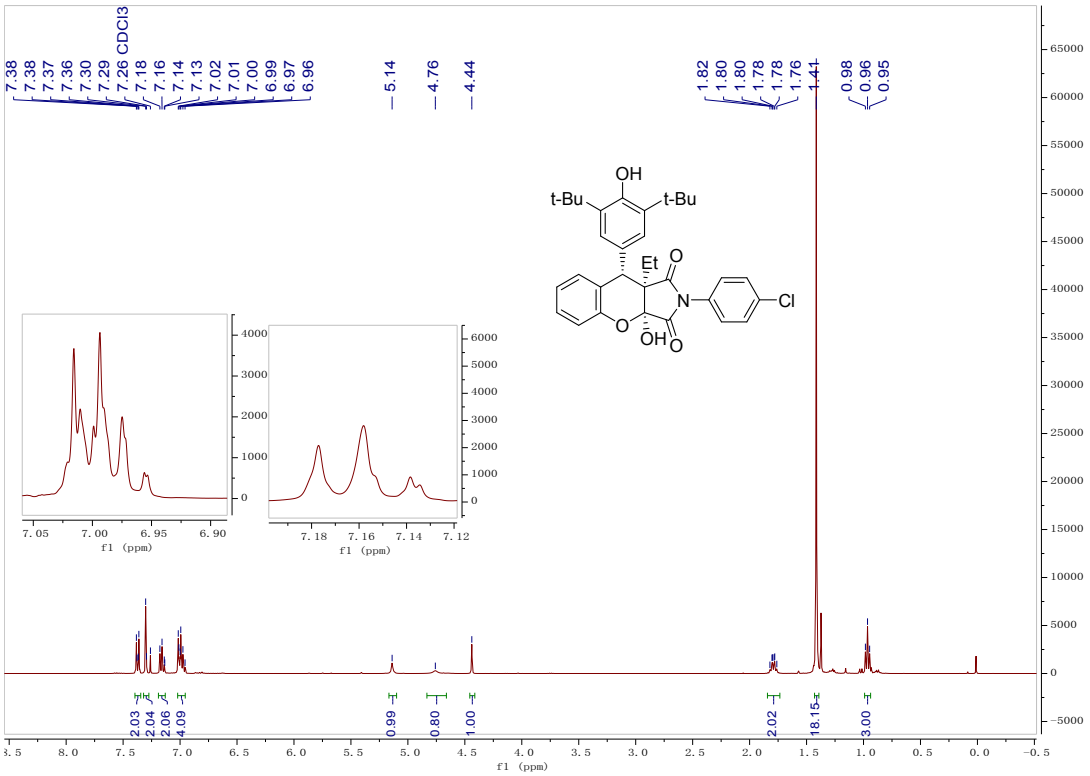


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.457	838.694	6771.564	1.0344
2	5.682	43818.391	647847.438	98.9656

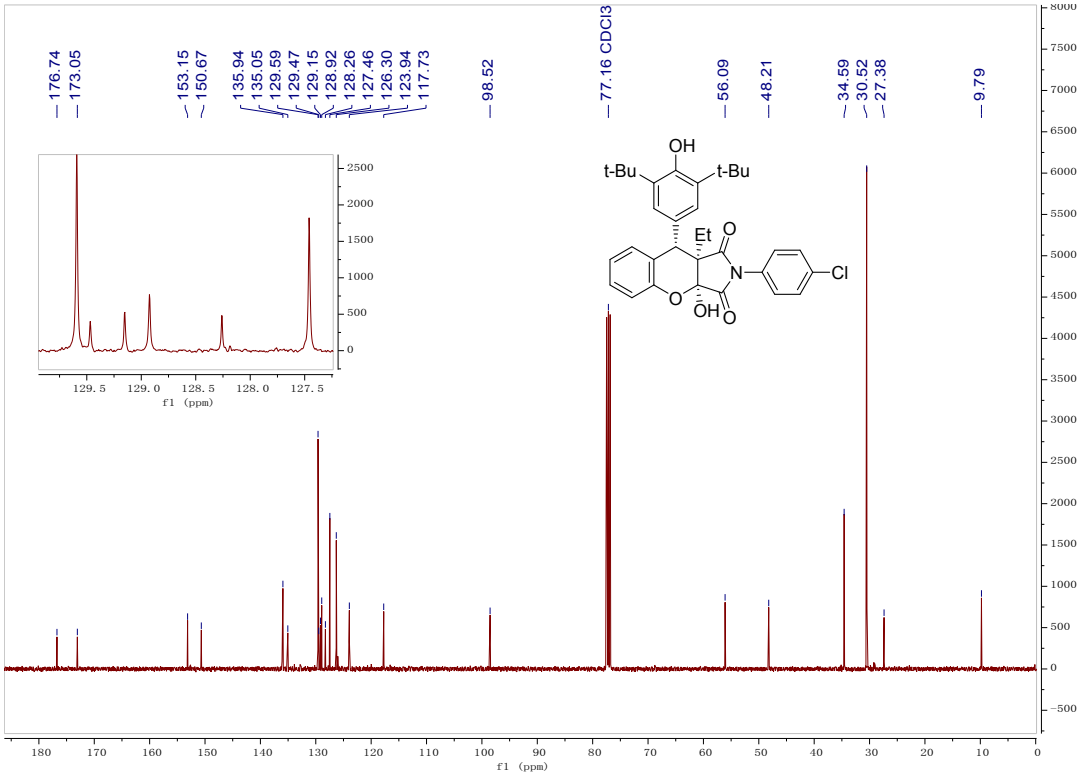
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.4 min, t_{major} = 5.6 min.

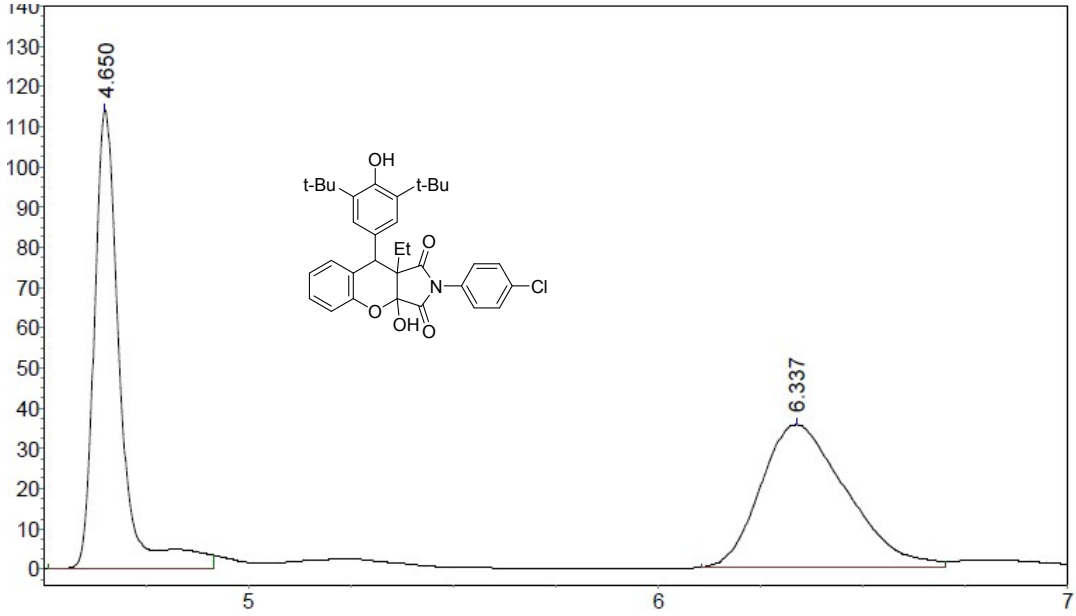
3ea

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

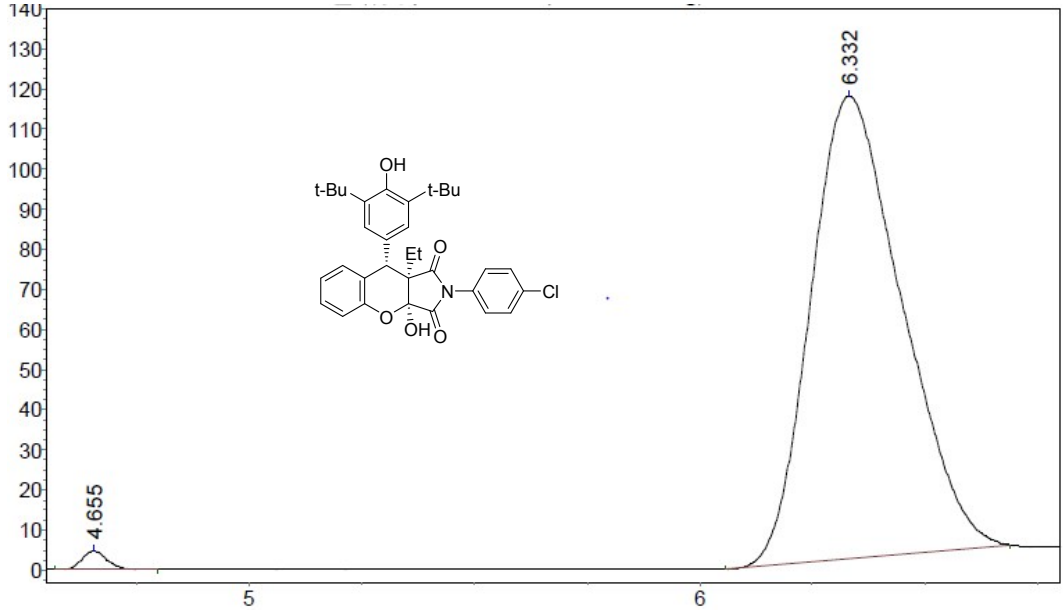


^{13}C NMR (100 MHz, $CDCl^3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.650	113818.000	499070.000	49.2184
2	6.337	35478.000	514919.875	50.7816

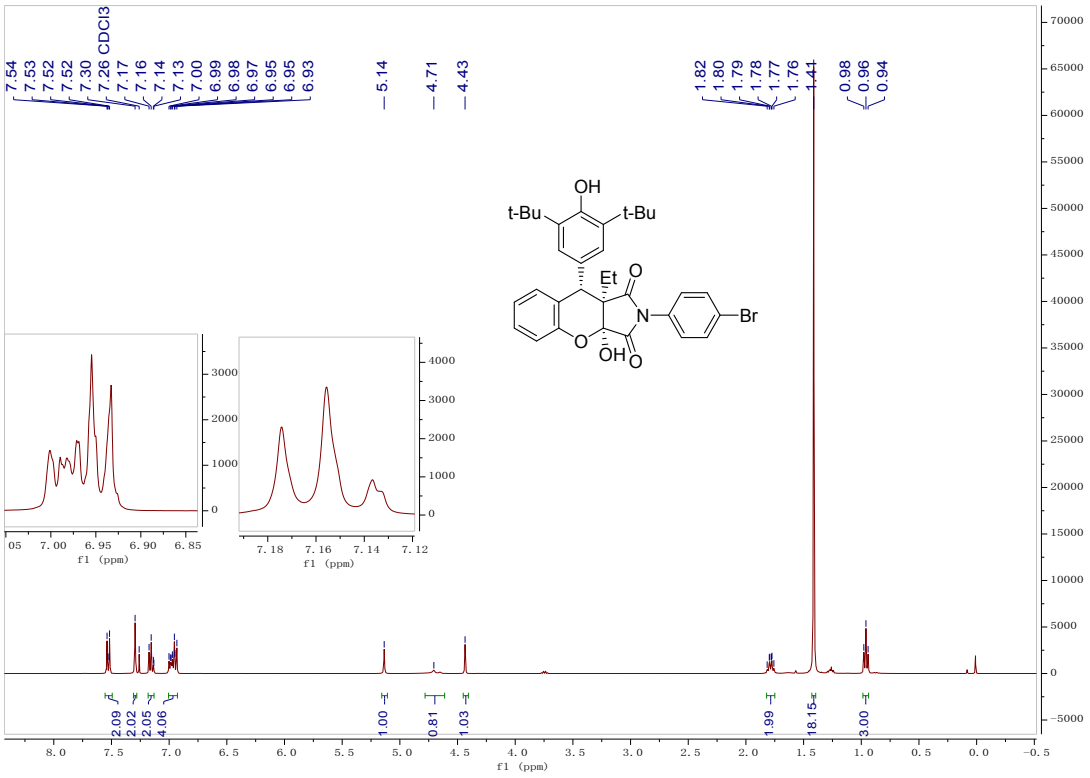


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.655	4683.115	18712.250	1.1440
2	6.332	115175.852	1617013.875	98.8560

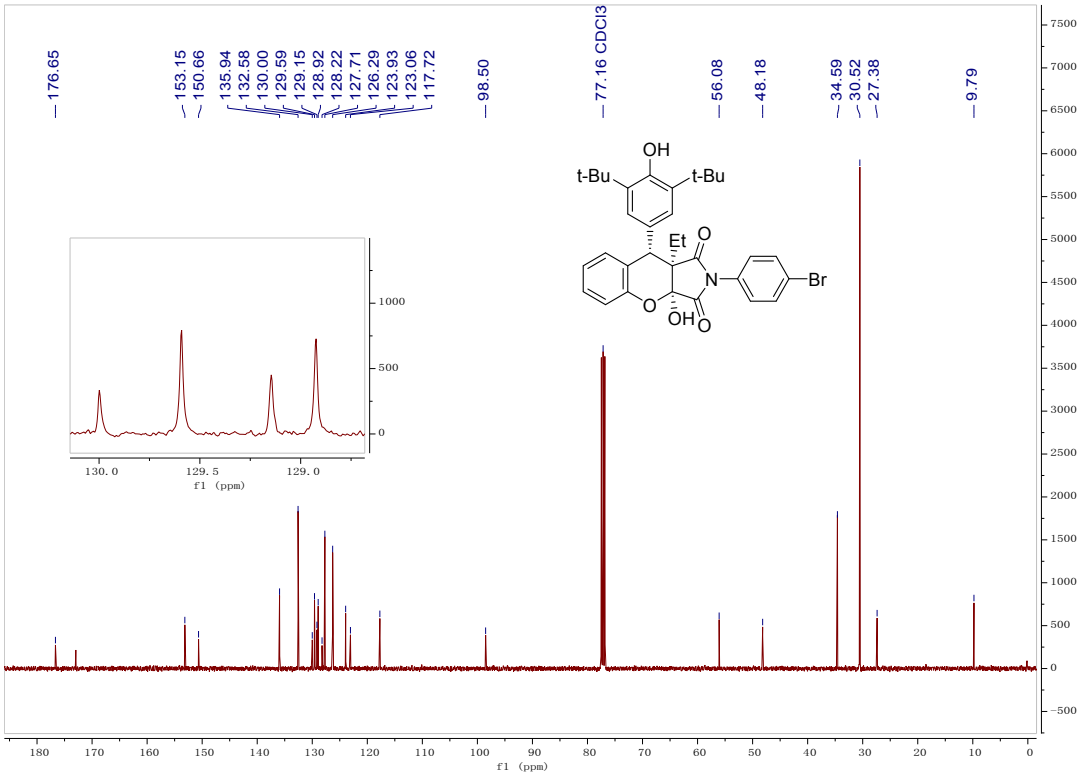
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.6 min, t_{major} = 6.3 min.

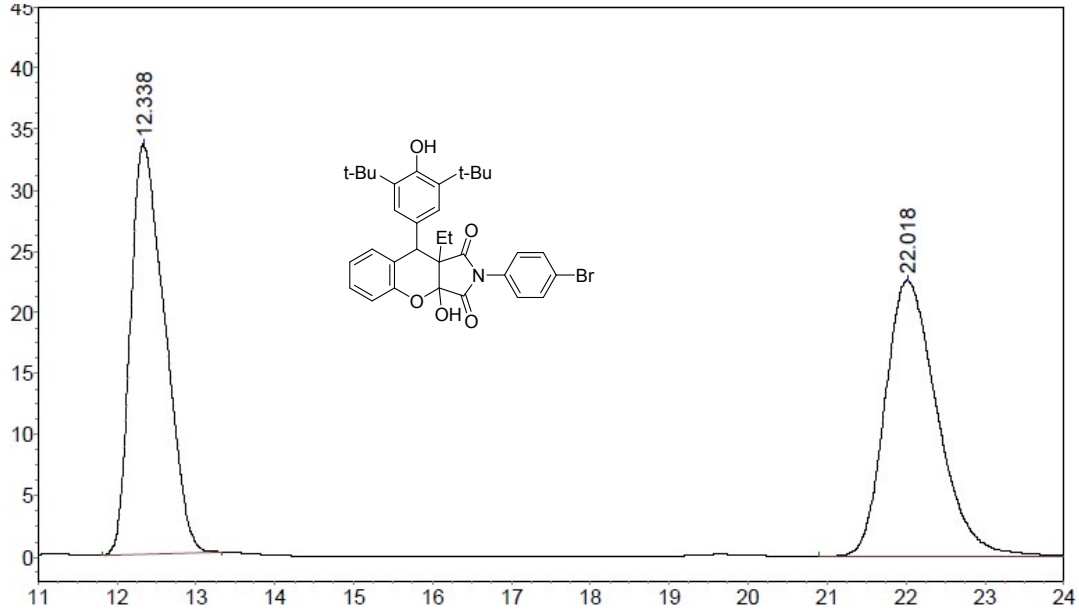
3fa

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

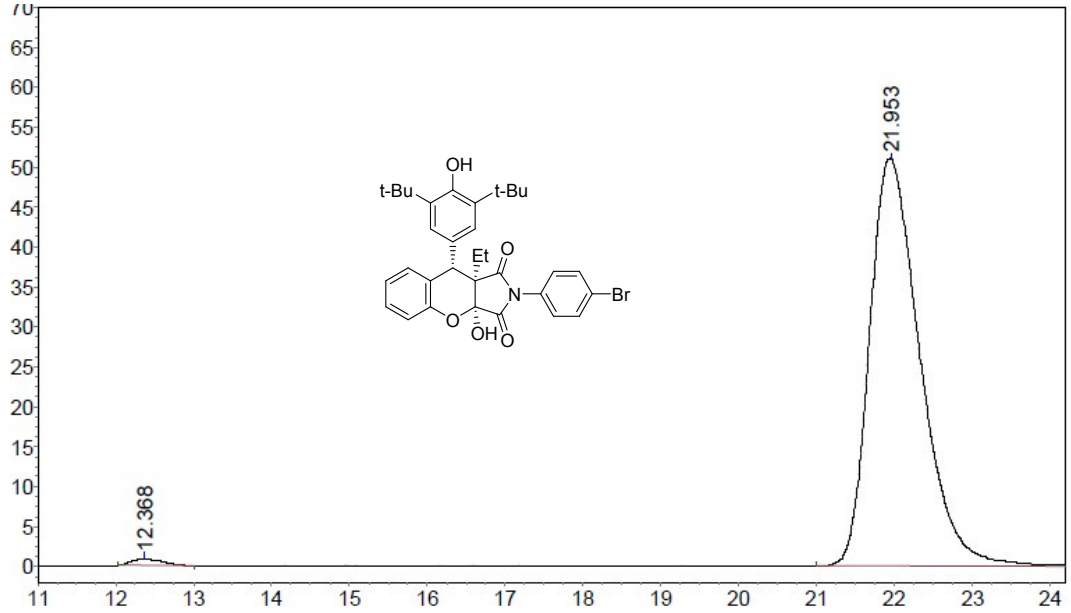


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	12.338	33573.477	1012681.250	49.4176
2	22.018	22563.738	1036552.188	50.5824

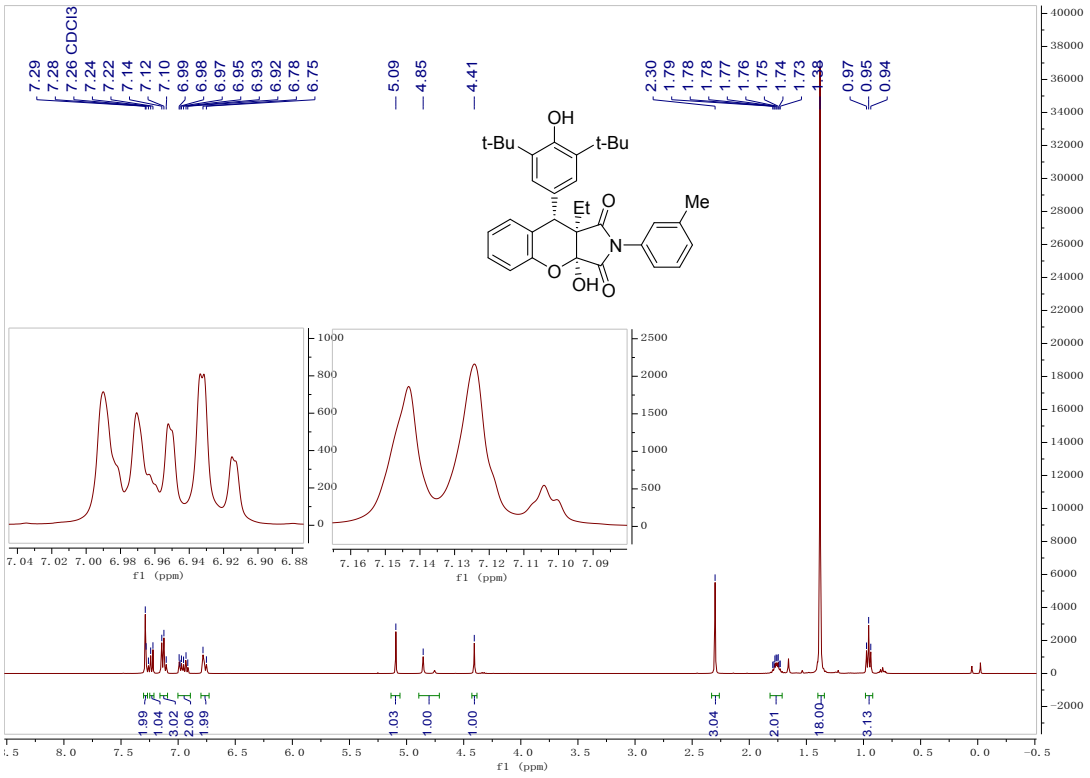


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	12.368	885.376	23706.900	1.0237
2	21.953	50985.473	2292195.250	98.9763

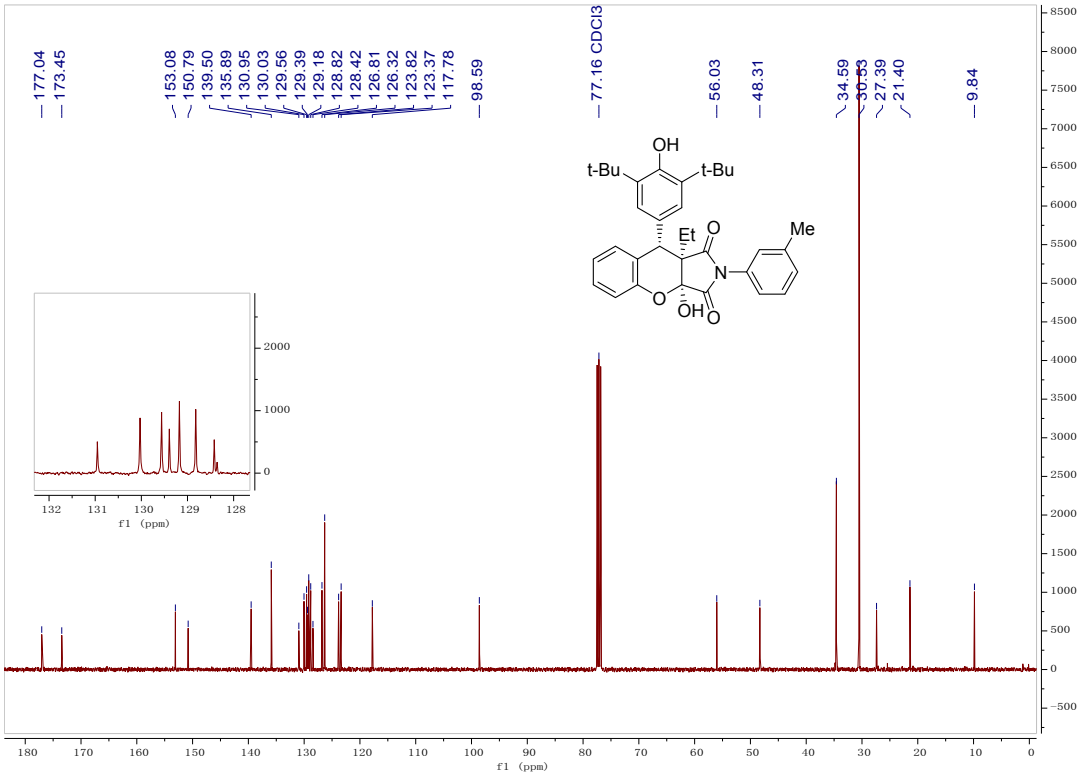
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 97/3, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 12.3 min, t_{major} = 21.9 min.

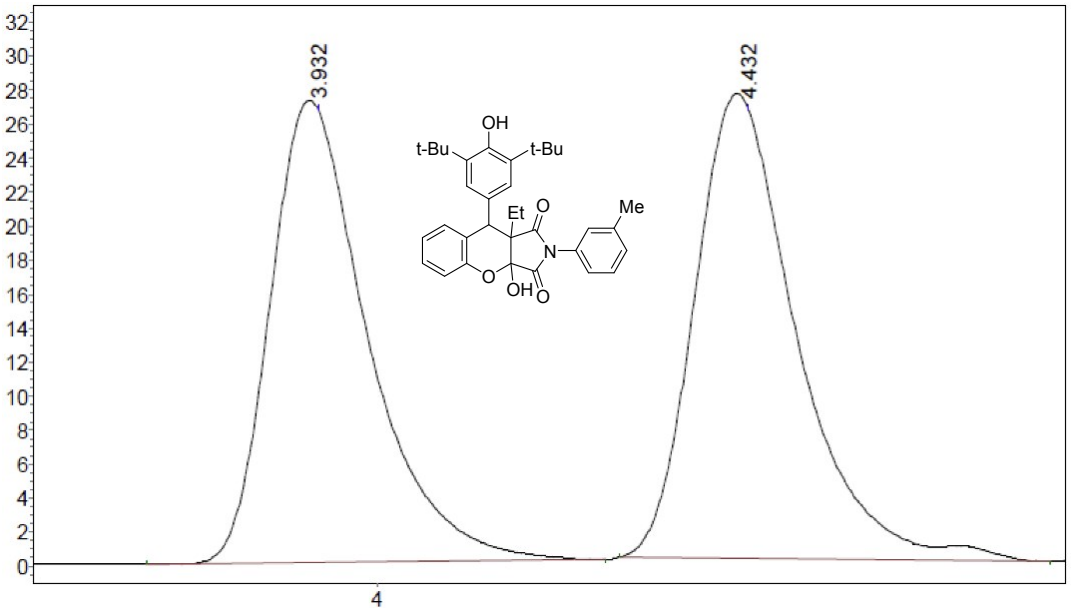
3ga

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

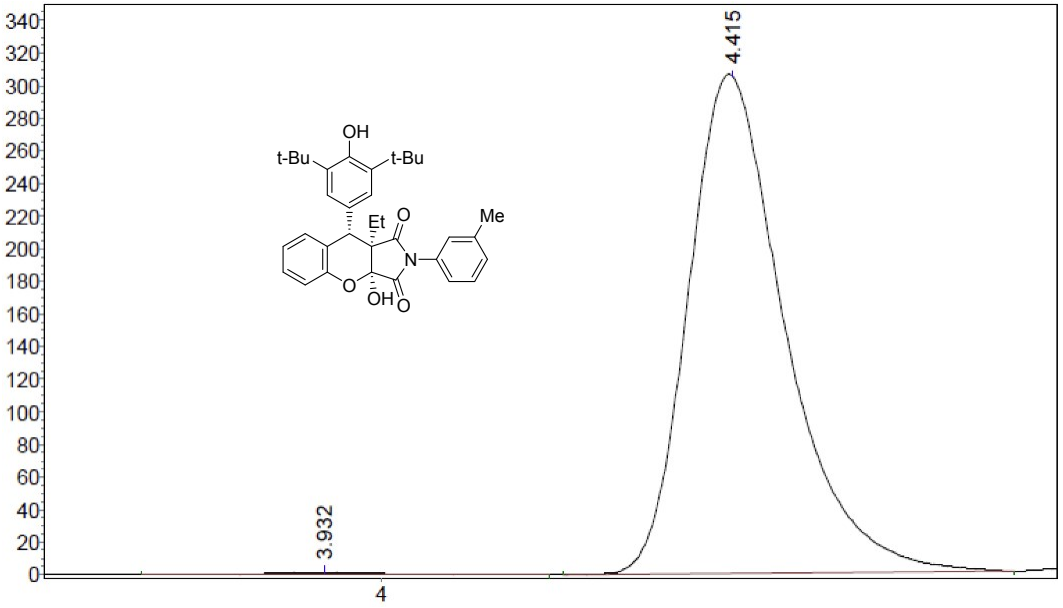


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	3.932	27014.750	213450.000	48.6847
2	4.432	27057.600	224983.297	51.3153

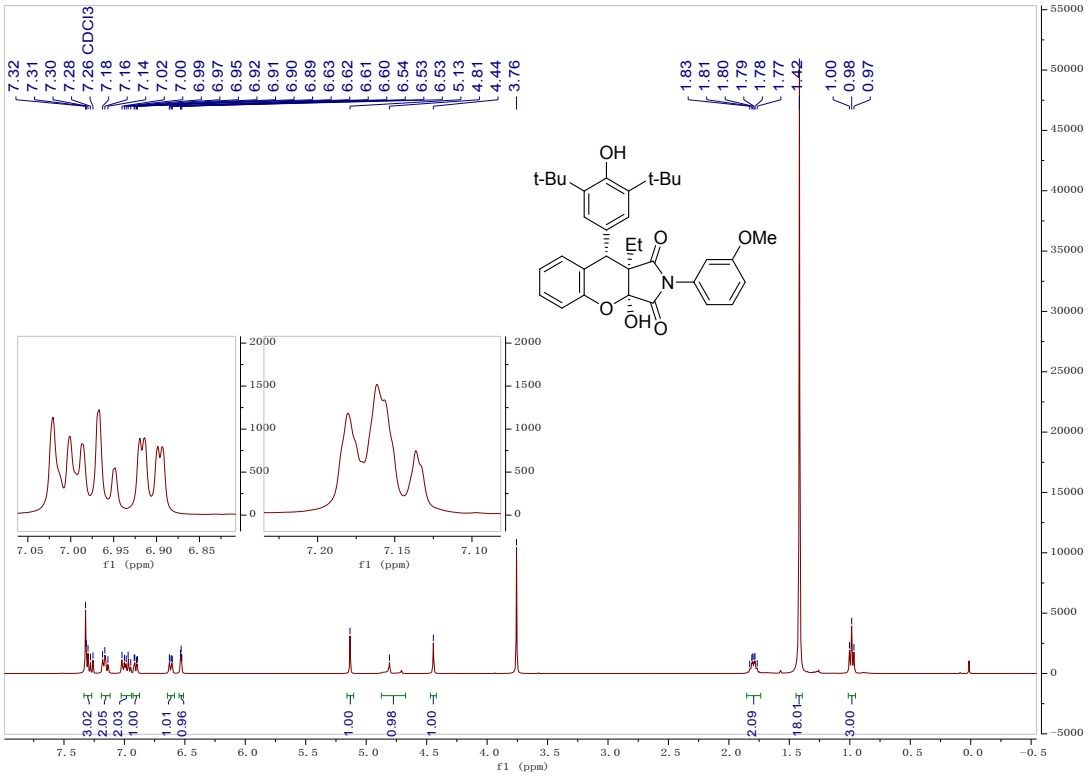


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	3.932	1550.586	11566.700	0.4760
2	4.415	303277.000	2418404.250	99.5240

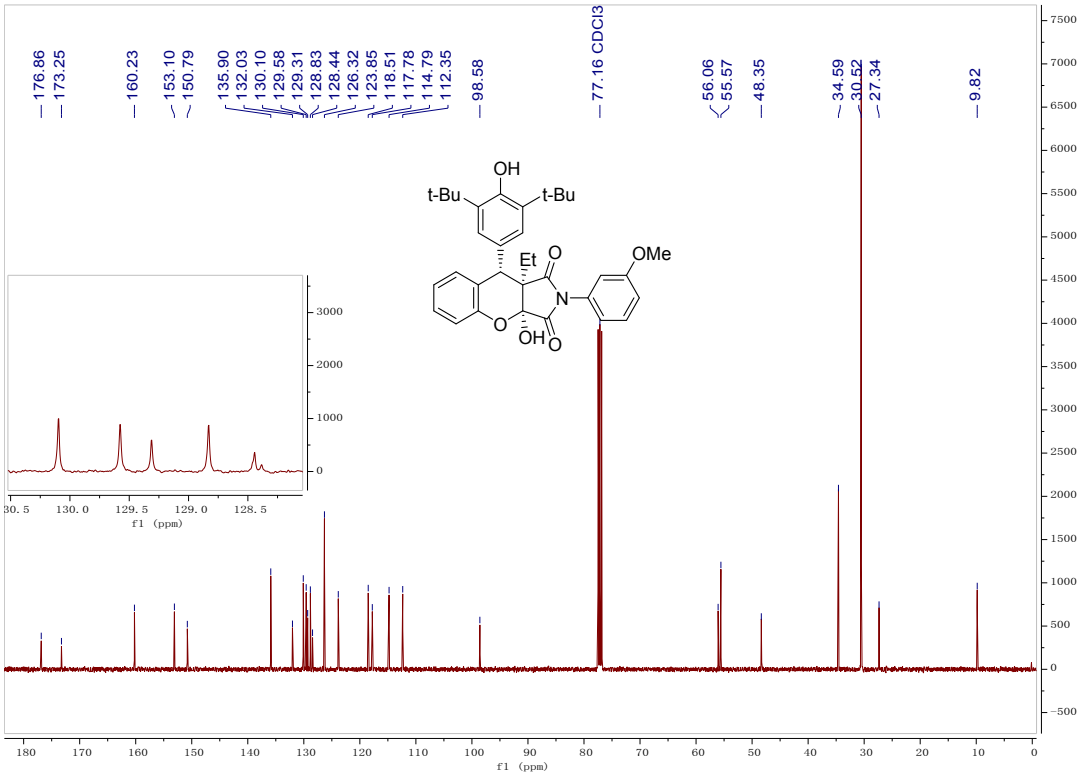
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 3.9 min, t_{major} = 4.4 min.

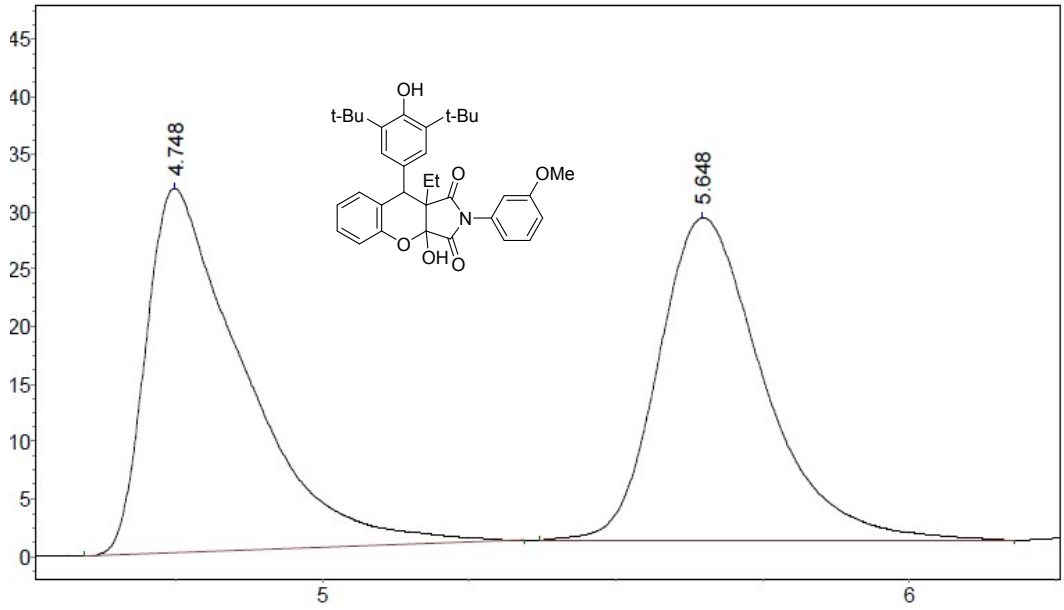
3ha

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

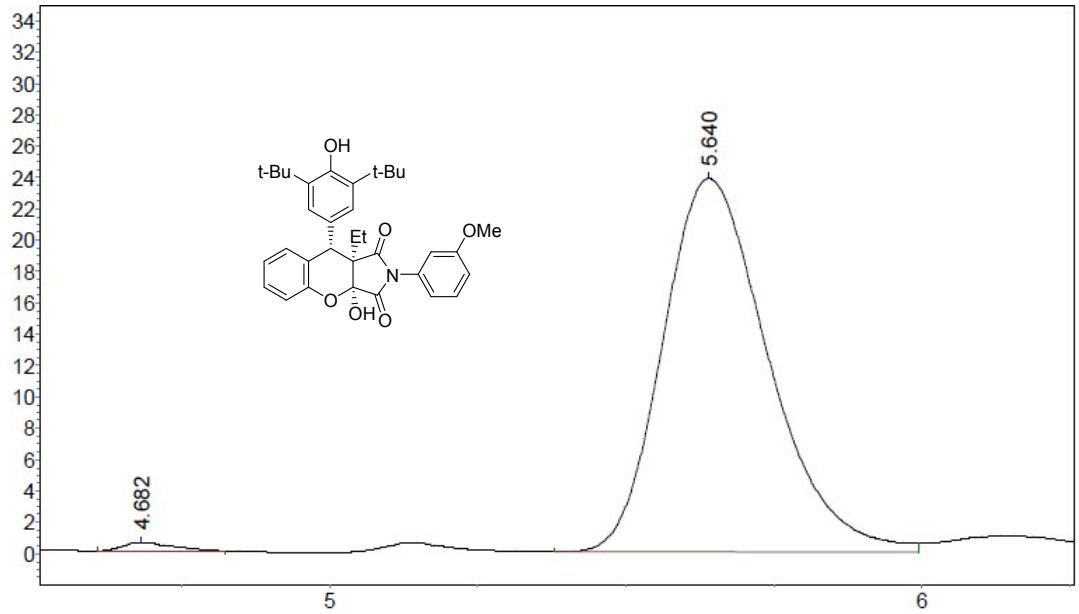


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.748	31661.867	369716.906	51.5560
2	5.648	28008.160	347400.188	48.4440

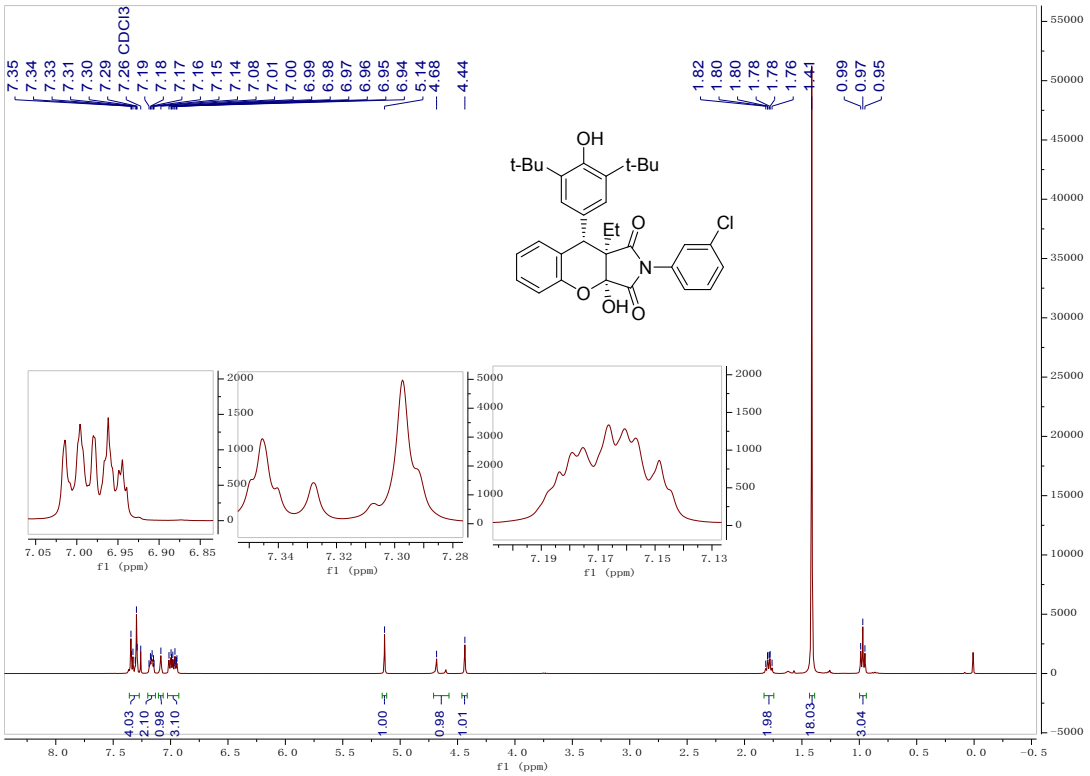


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.682	523.328	3095.500	1.0378
2	5.640	23865.783	295166.531	98.9622

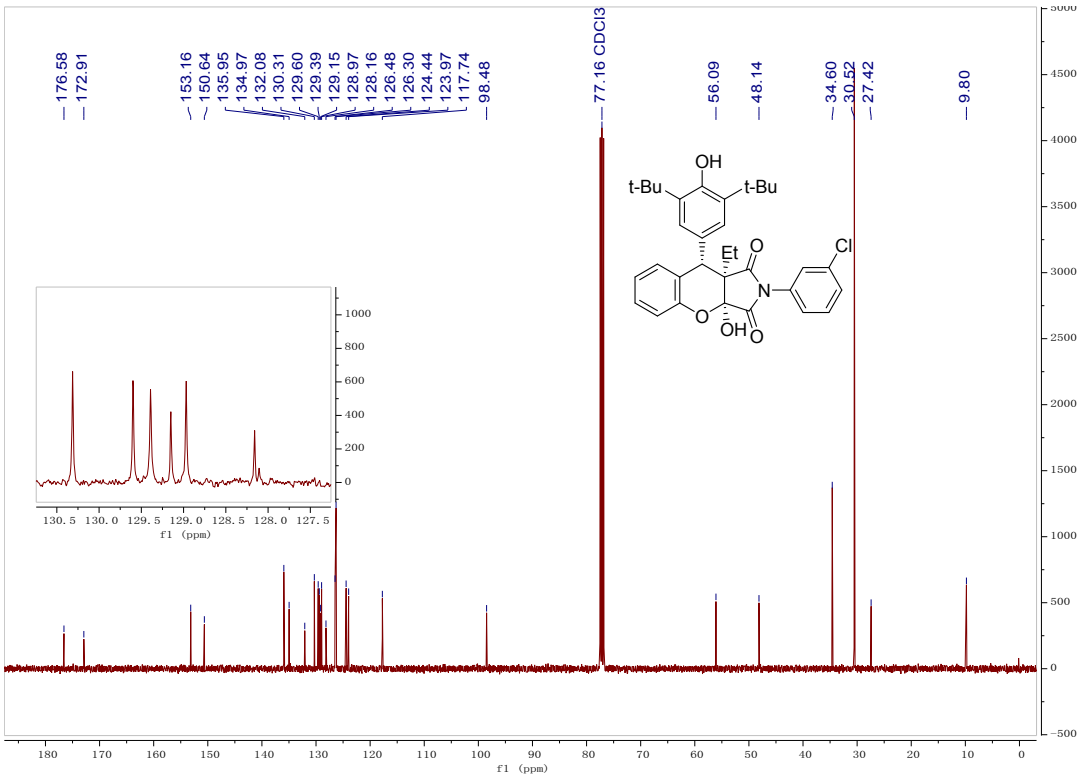
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 97/3, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.6 min, t_{major} = 5.6 min.

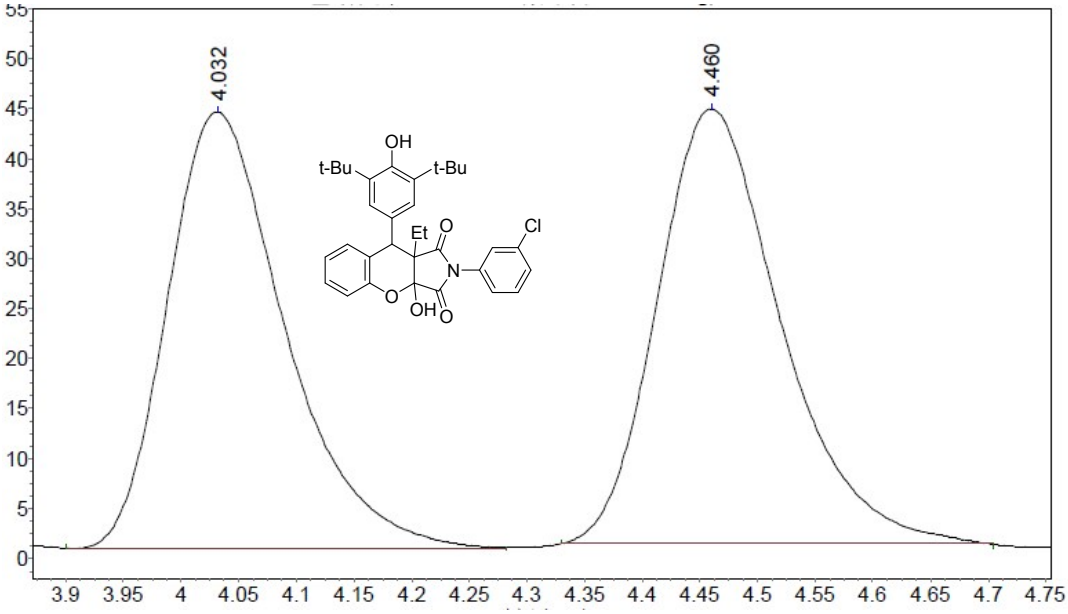
3ia

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

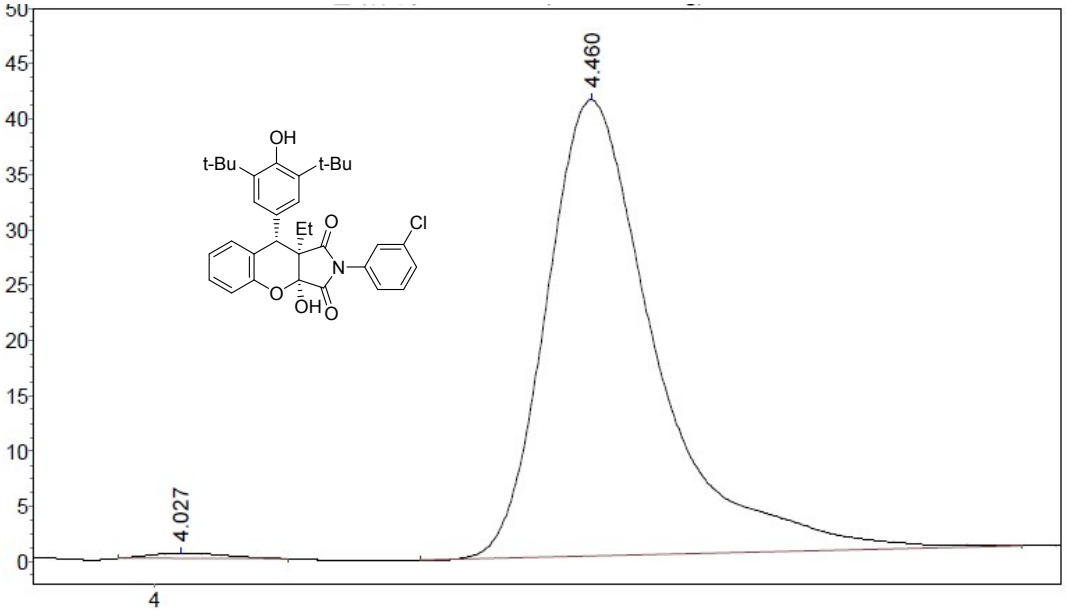


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.032	43593.430	307118.000	48.7452
2	4.460	43460.547	322929.594	51.2548

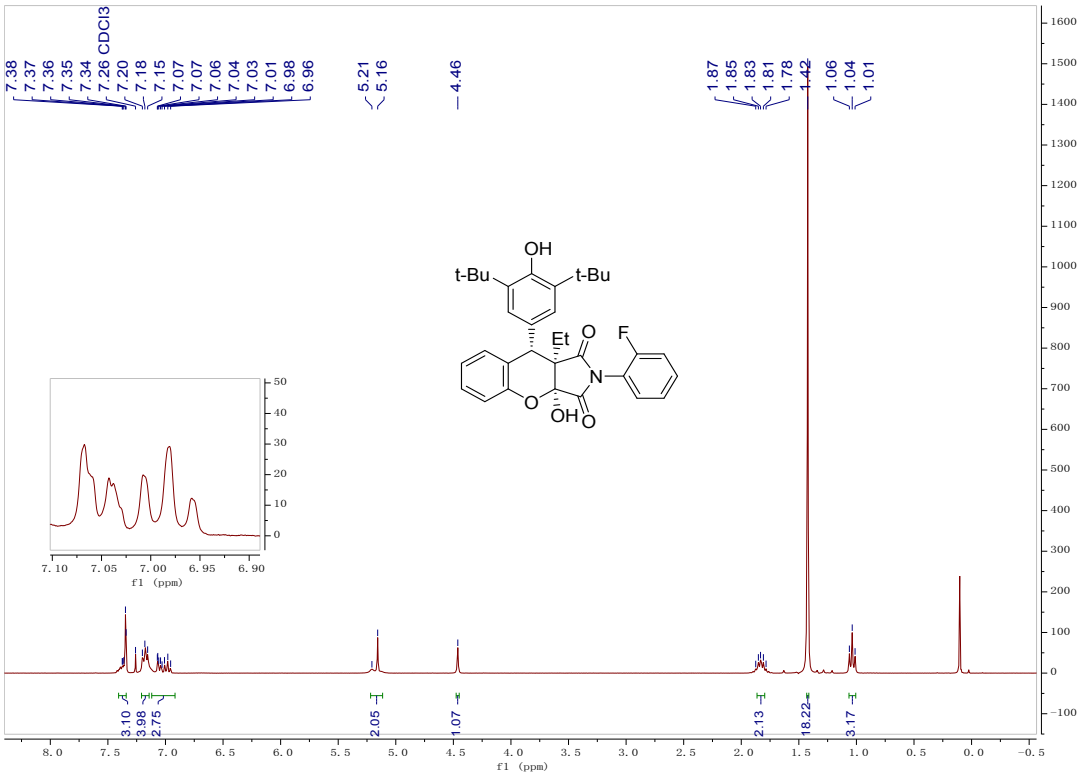


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.027	483.871	2708.541	0.8130
2	4.460	41220.000	330457.969	99.1870

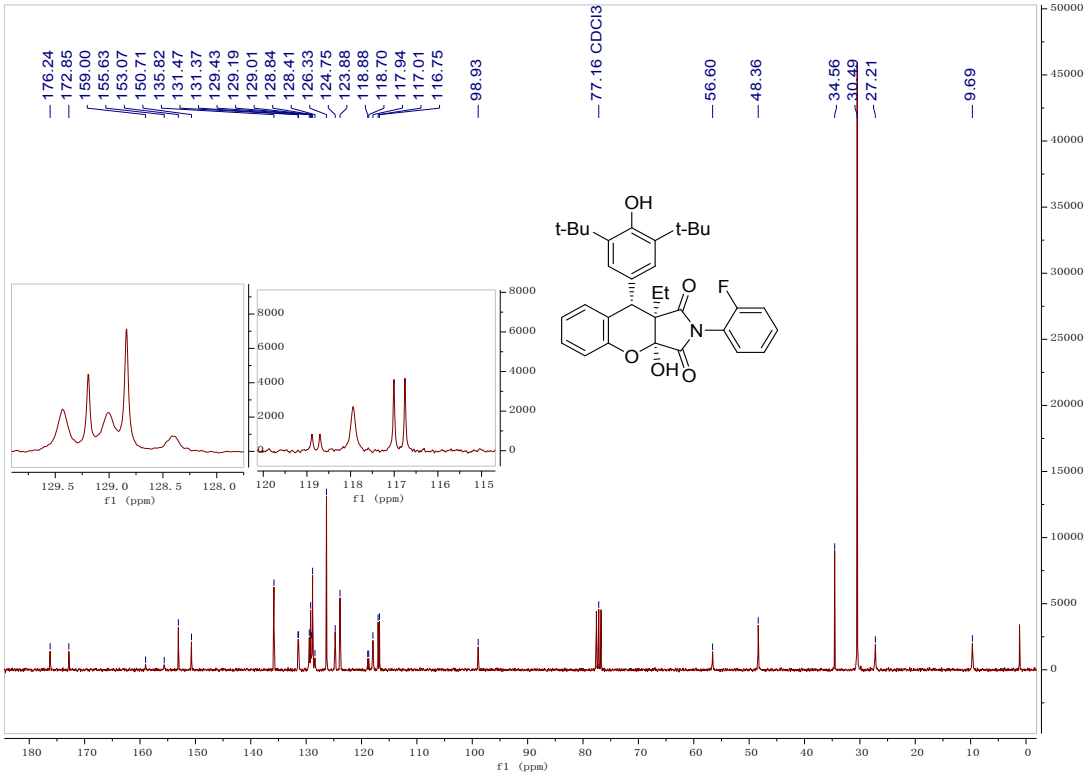
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ= 254 nm, t_{minor} = 4.0 min, t_{major} = 4.4 min

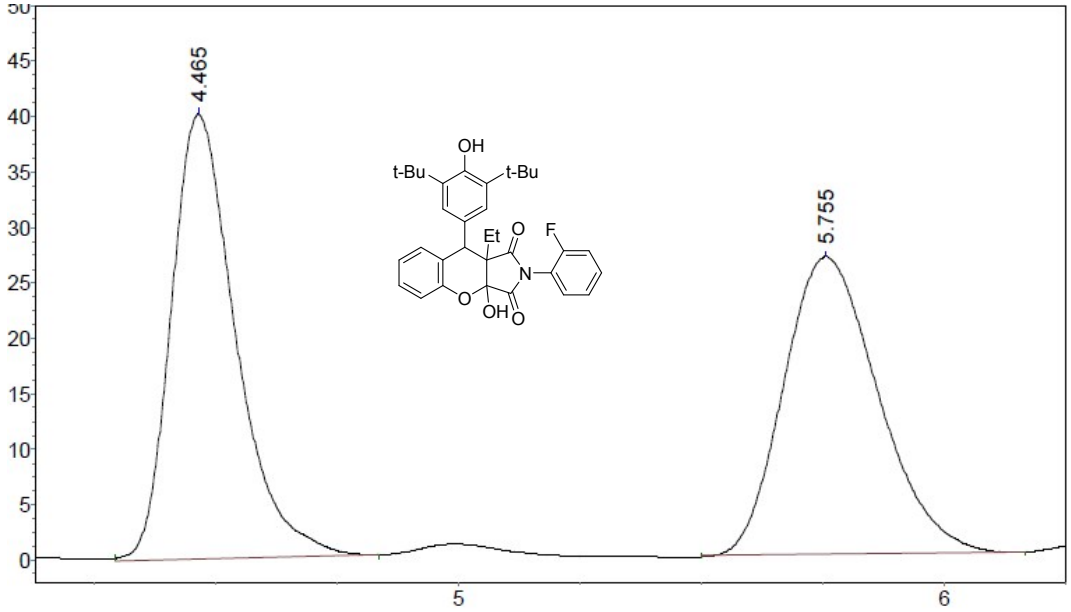
3ja

^{1}H NMR (300 MHz, $CDCl_3$)

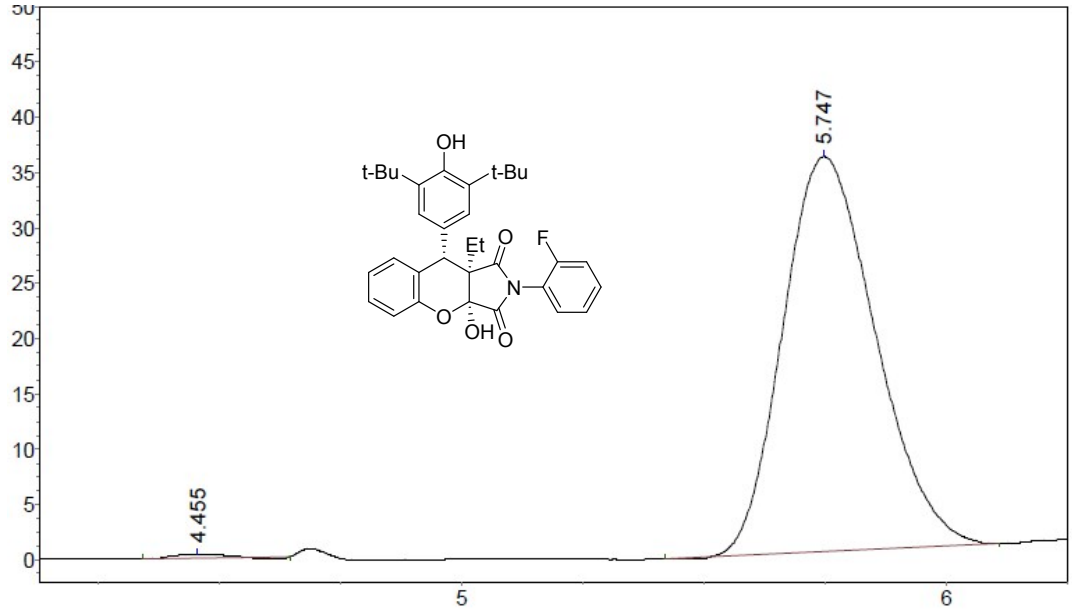


^{13}C NMR (700 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.465	40138.246	370628.313	50.6426
2	5.755	26817.678	361222.969	49.3574

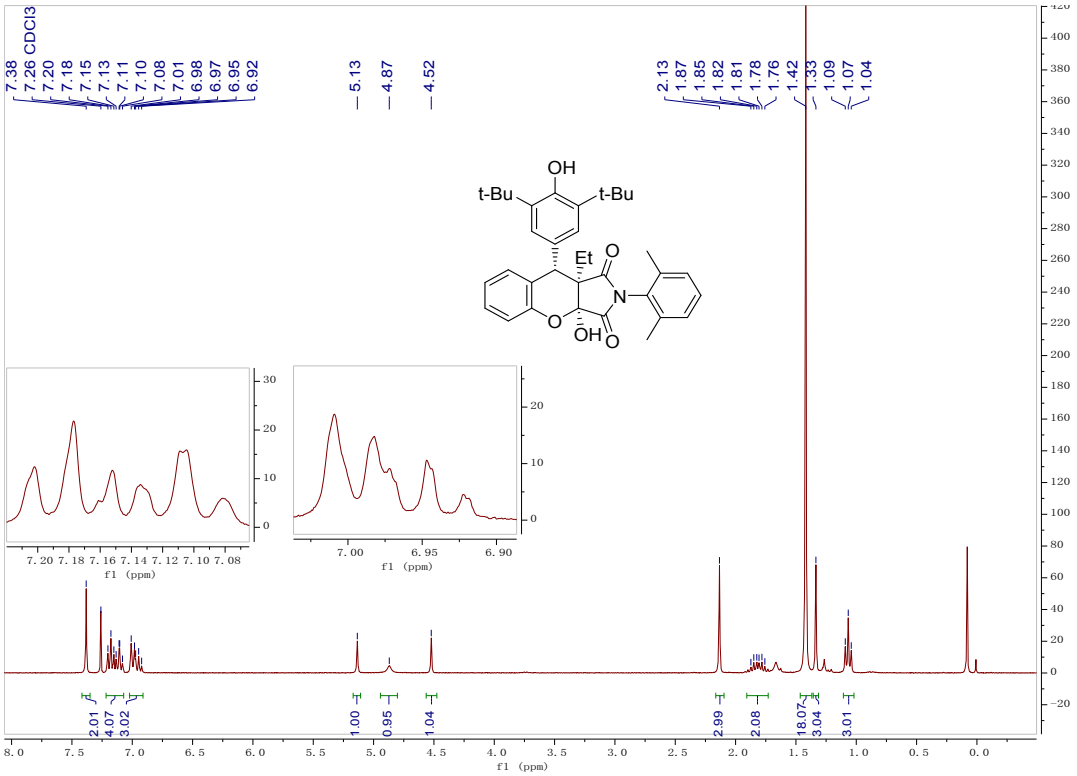


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.455	350.879	2200.800	0.4634
2	5.747	35664.031	472678.000	99.5366

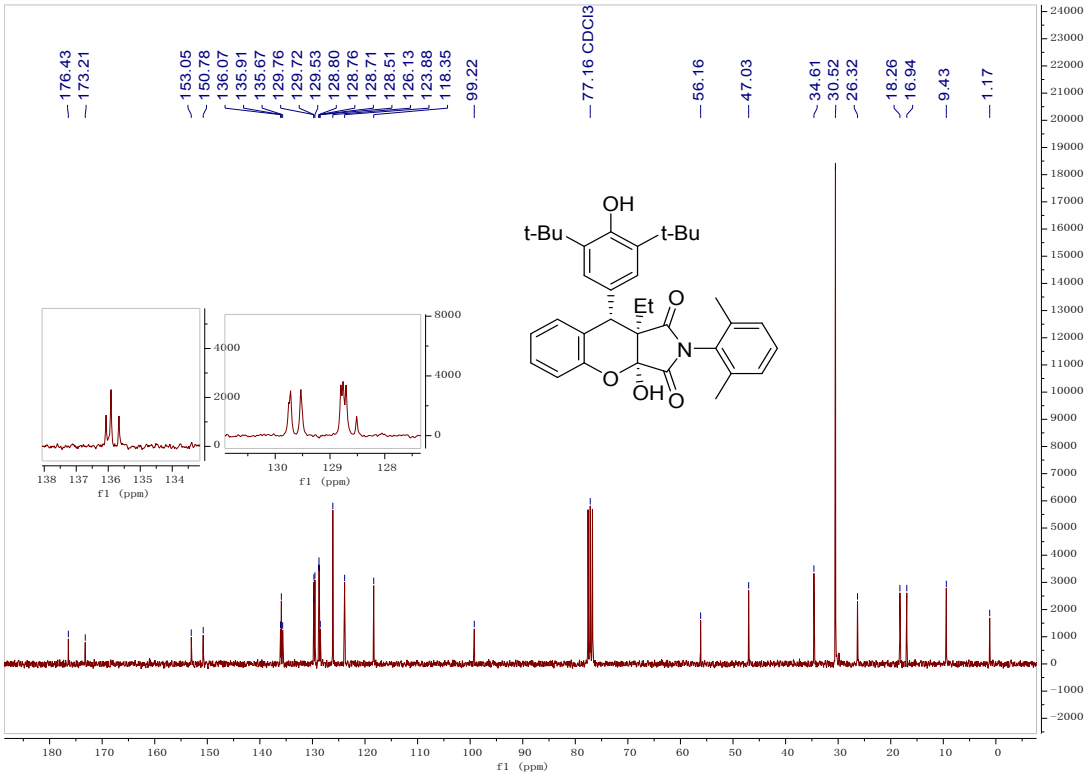
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.4 min, t_{major} = 5.7 min.

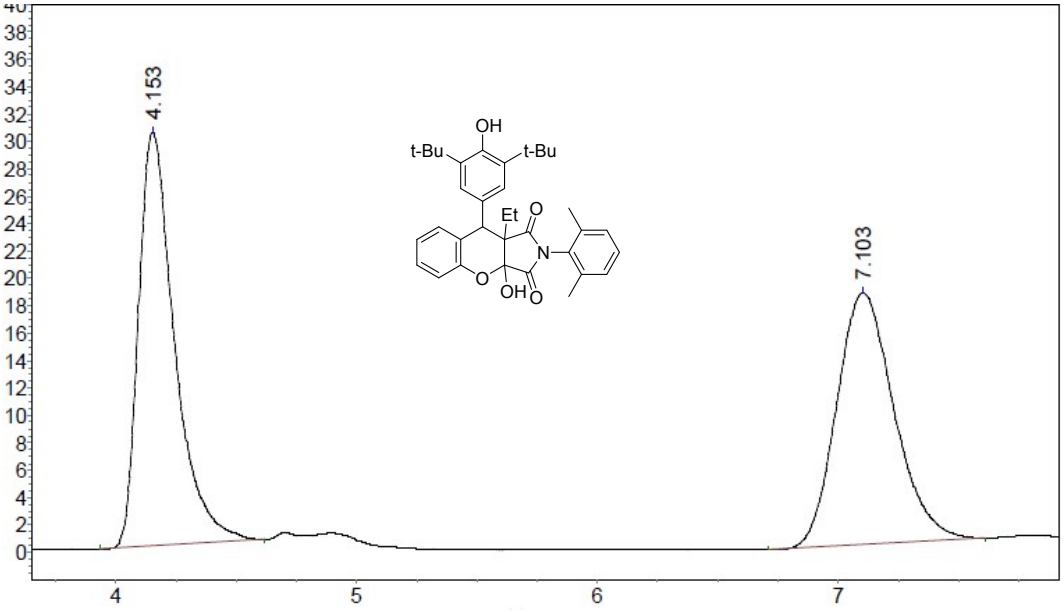
3ka

^{1}H NMR (300 MHz, $CDCl_3$)

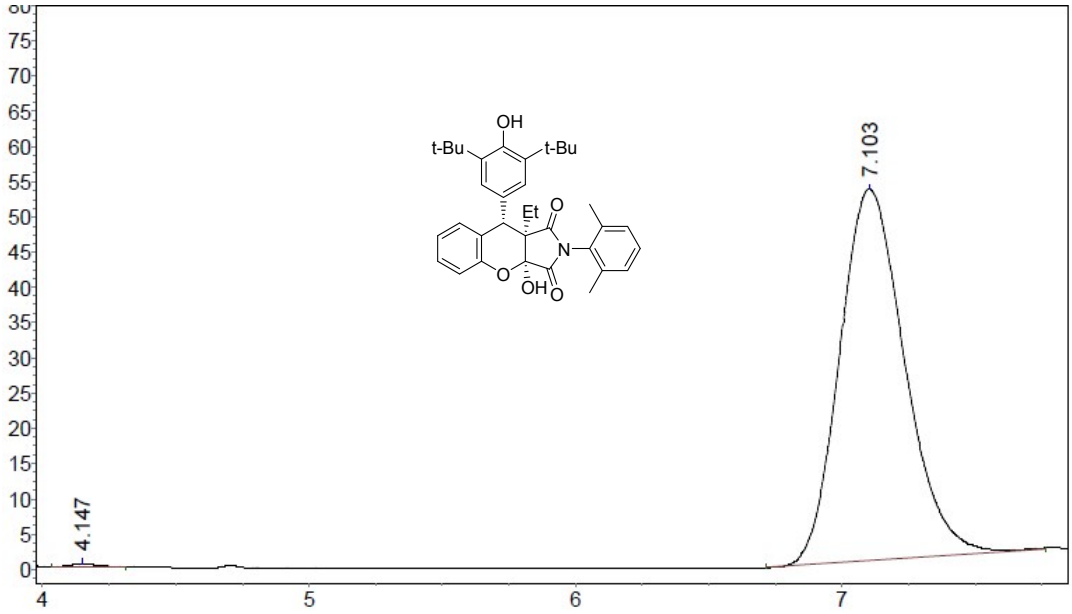


^{13}C NMR (75 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.153	30148.496	312959.844	50.7090
2	7.103	18365.838	304207.906	49.2910

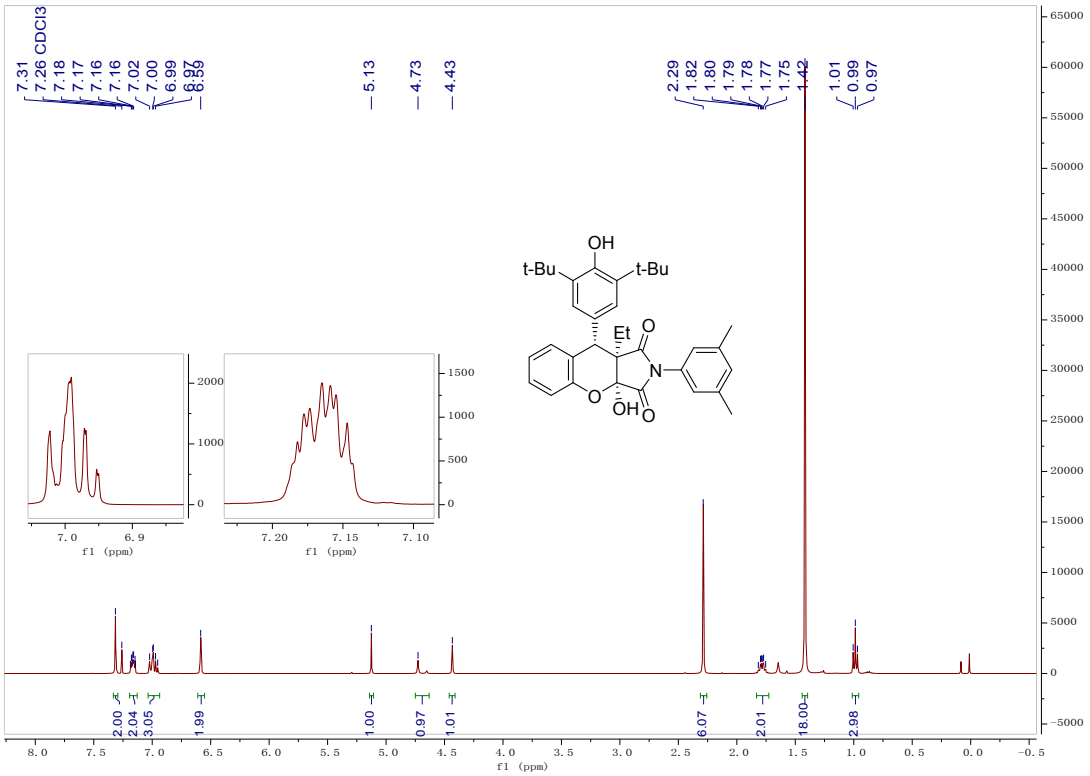


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.147	411.757	3230.000	0.3607
2	7.103	52694.594	892355.625	99.6393

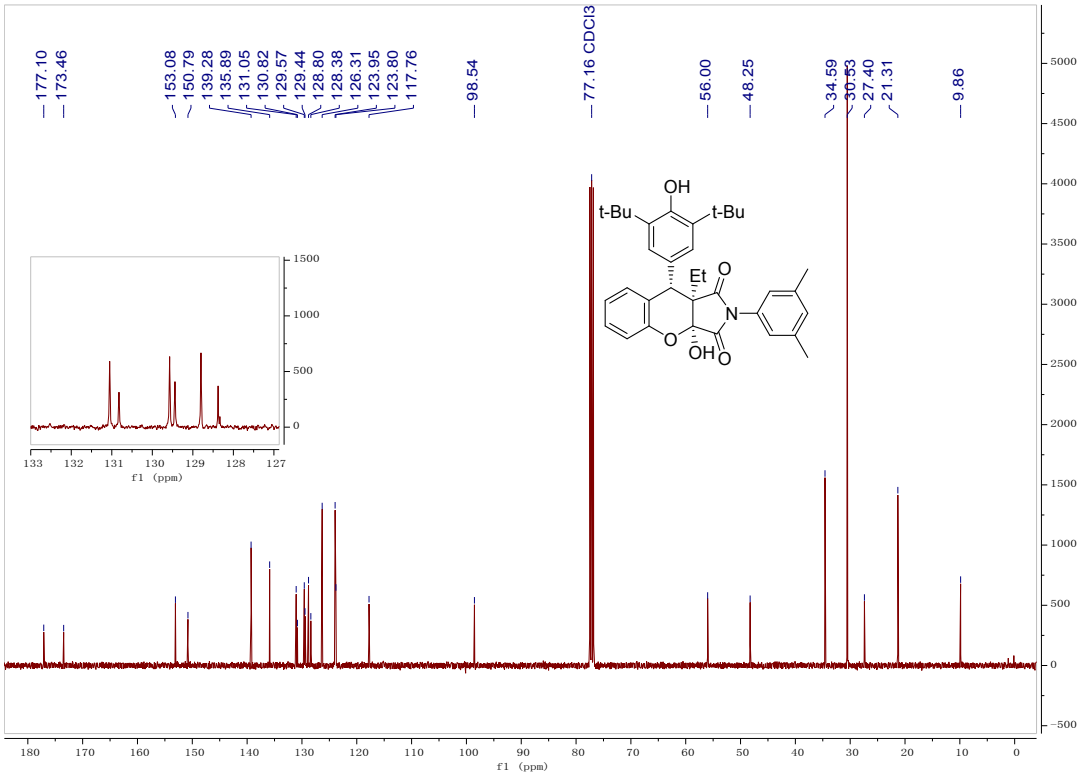
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.1 min, t_{major} = 7.1 min.

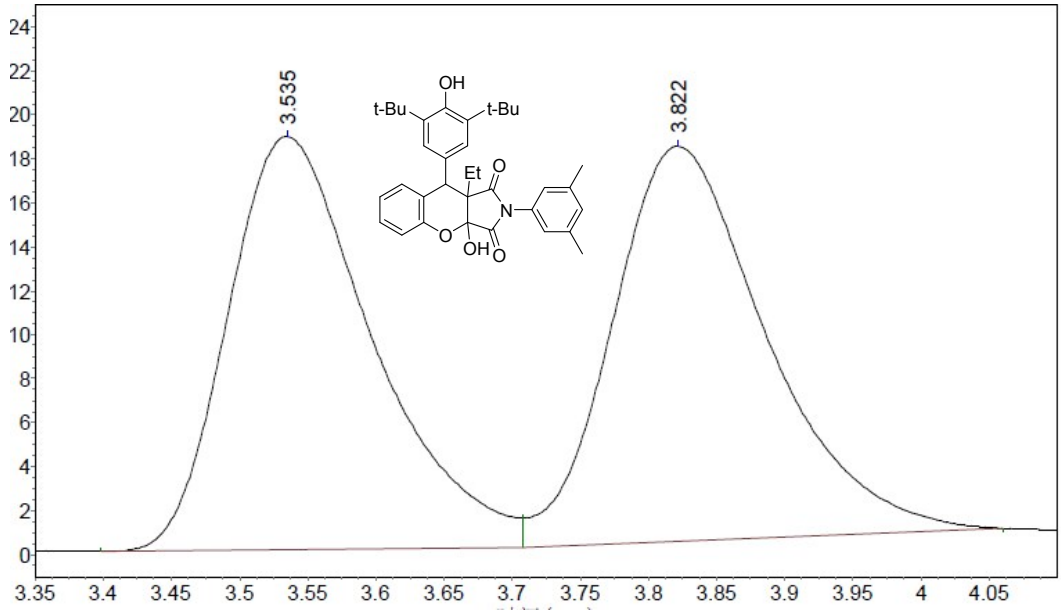
3la

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

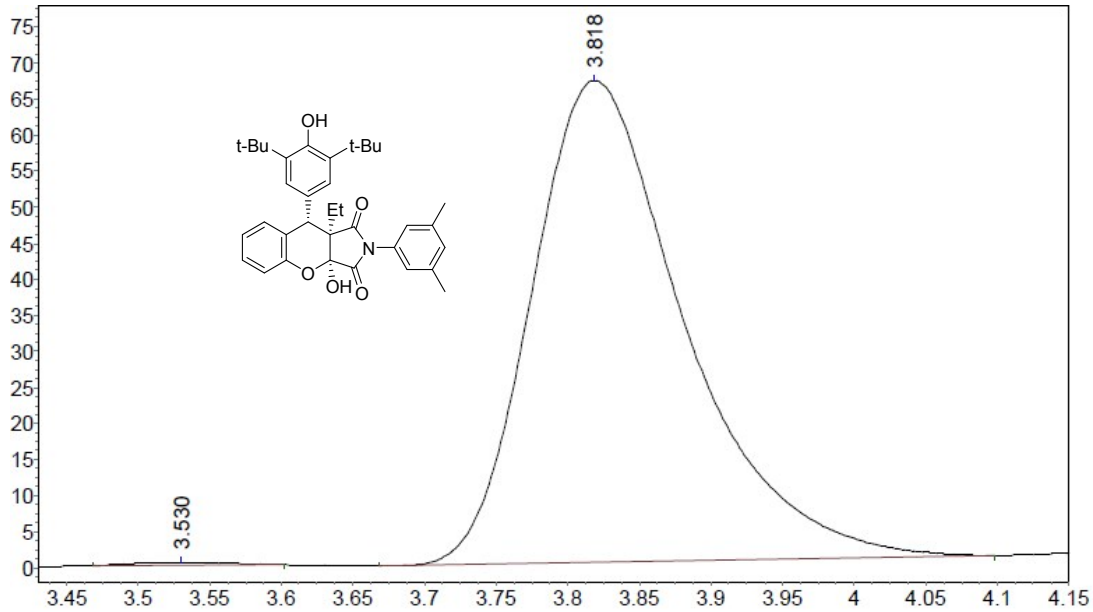


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	3.535	18770.768	138422.531	49.4914
2	3.822	17948.994	141267.625	50.5086

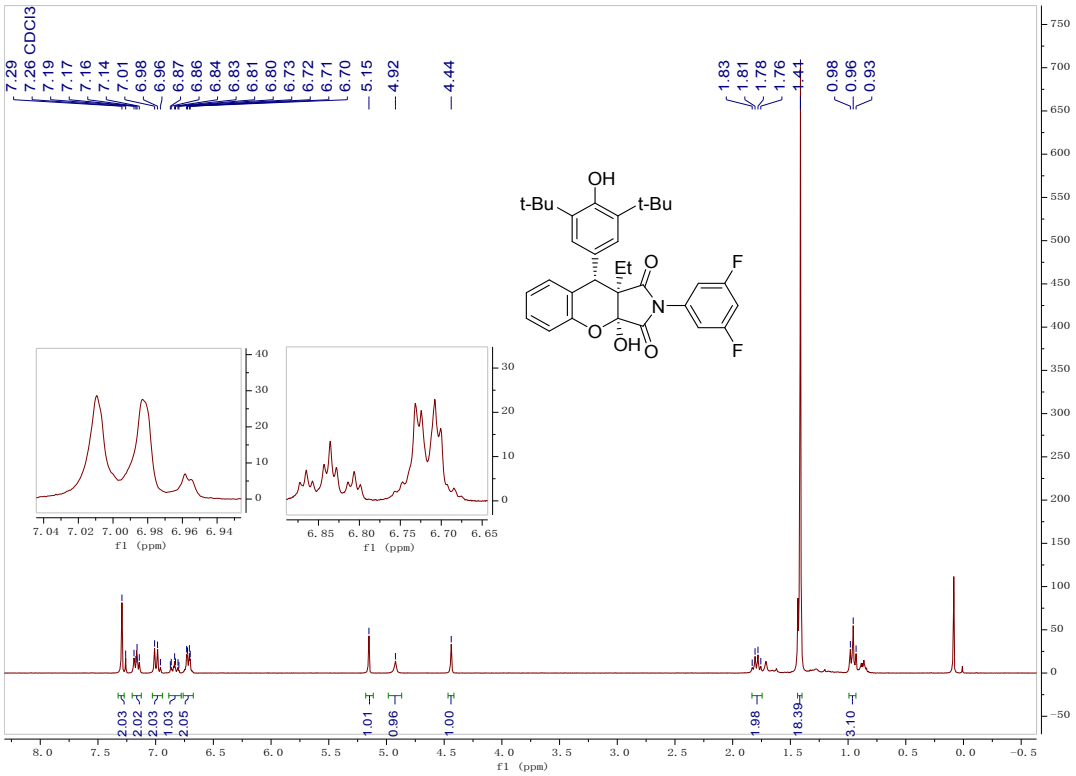


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	3.530	414.494	1888.300	0.3809
2	3.818	66869.219	493879.281	99.6191

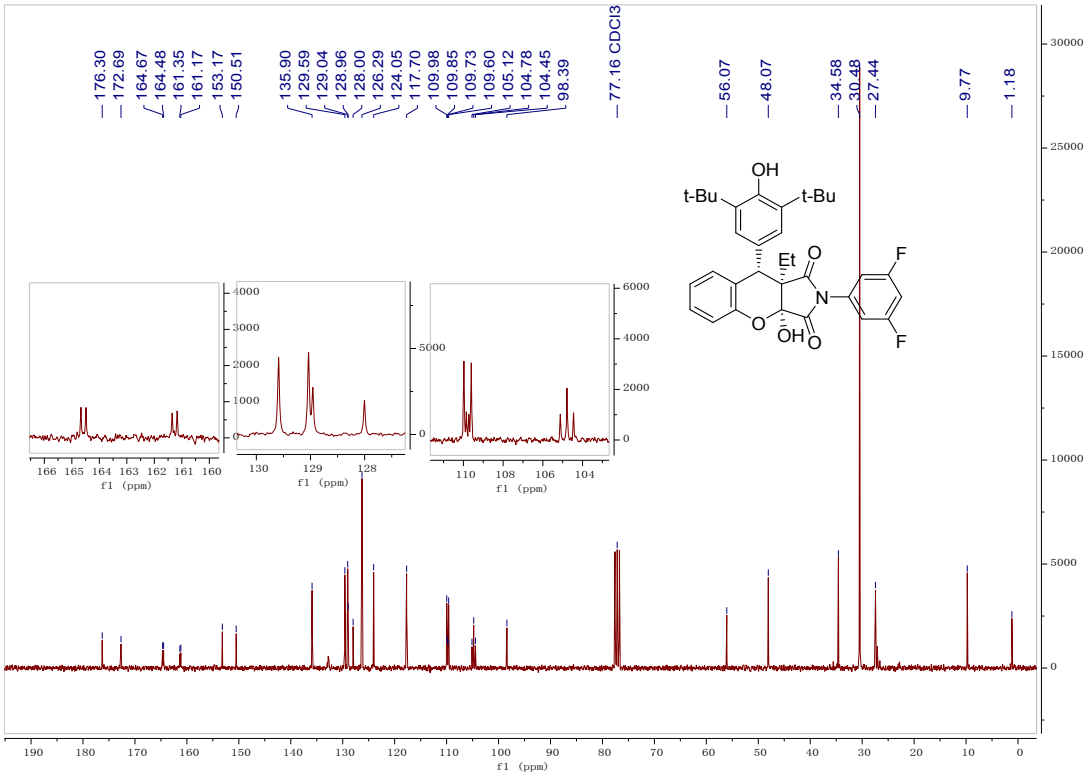
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 3.5 min, t_{major} = 3.8 min

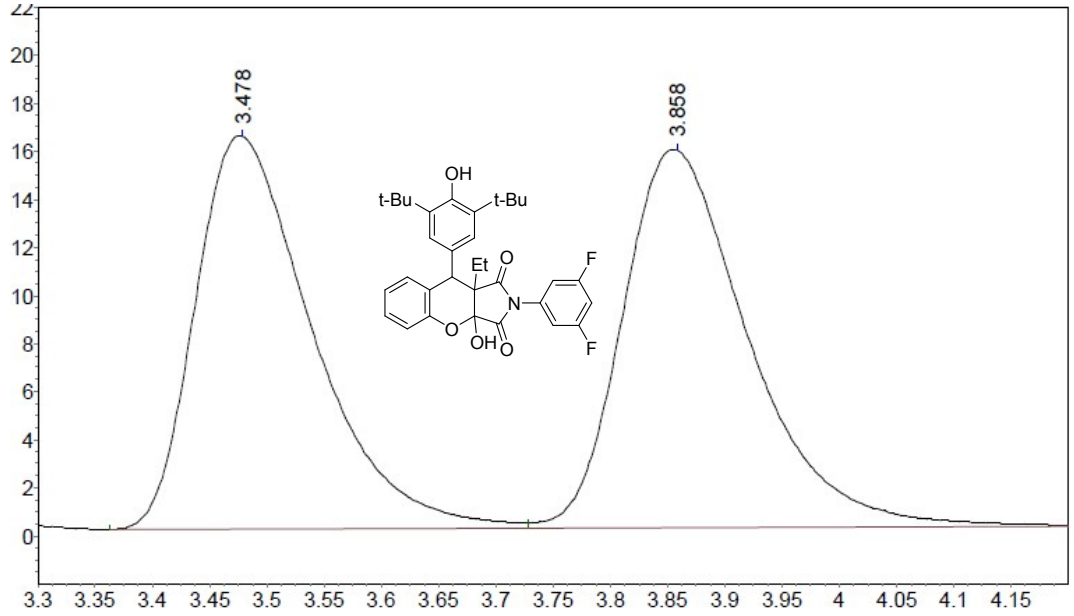
3ma

^{1}H NMR (300 MHz, $CDCl_3$)

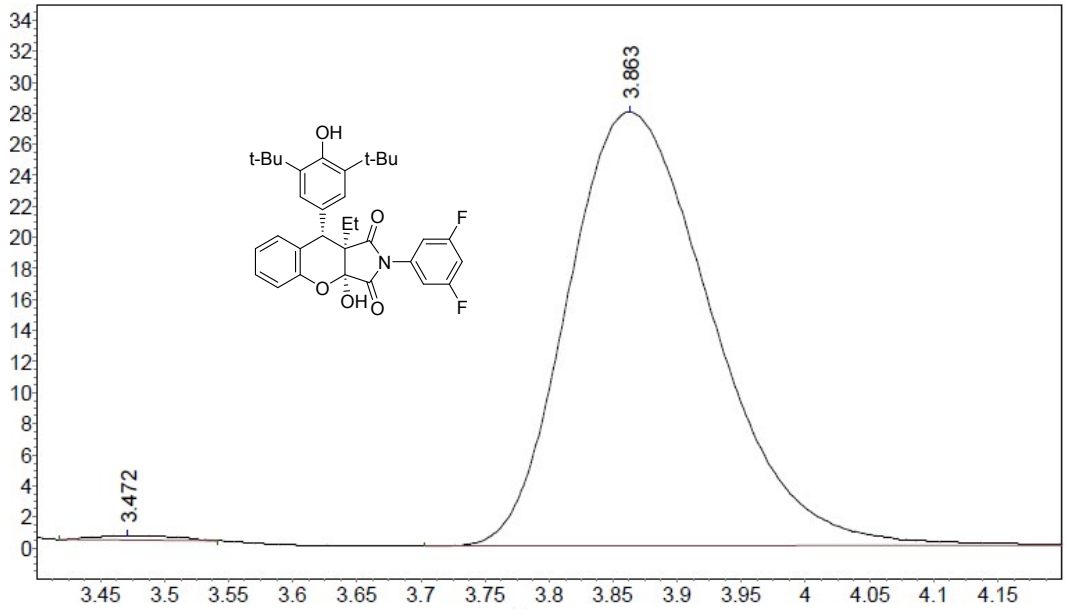


^{13}C NMR (75 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	3.478	16364.178	116045.266	49.0372
2	3.858	15748.200	120602.234	50.9628

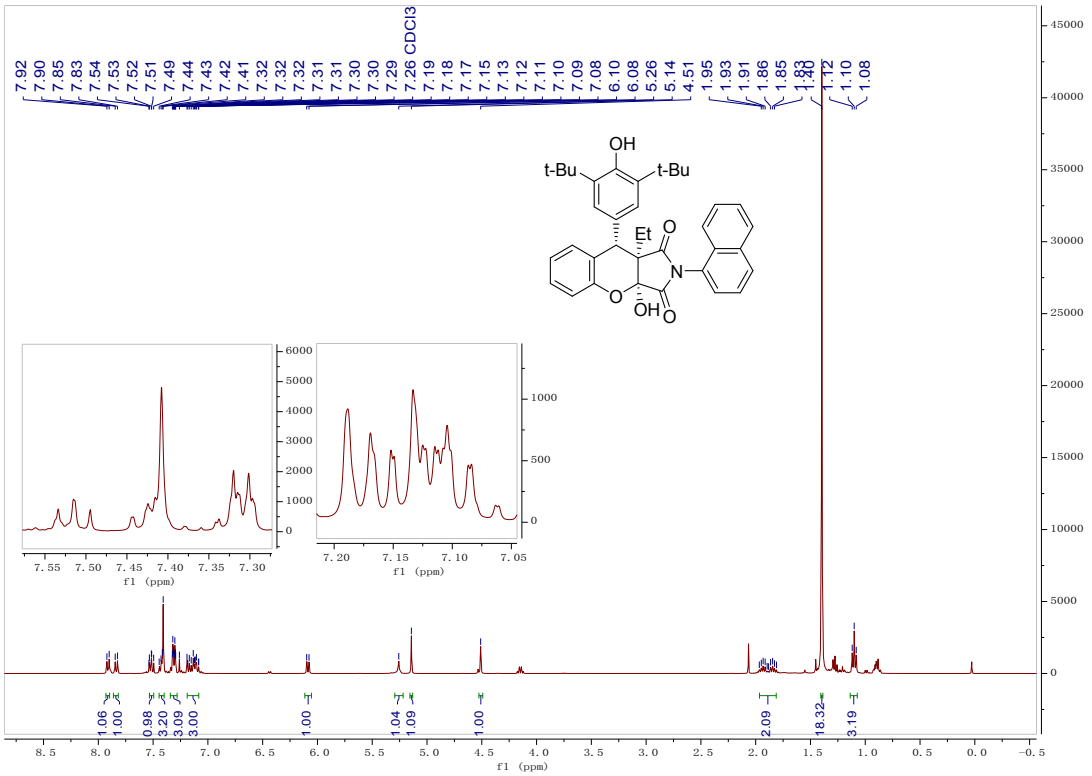


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	3.472	243.680	1066.550	0.4845
2	3.863	27980.629	219054.359	99.5155

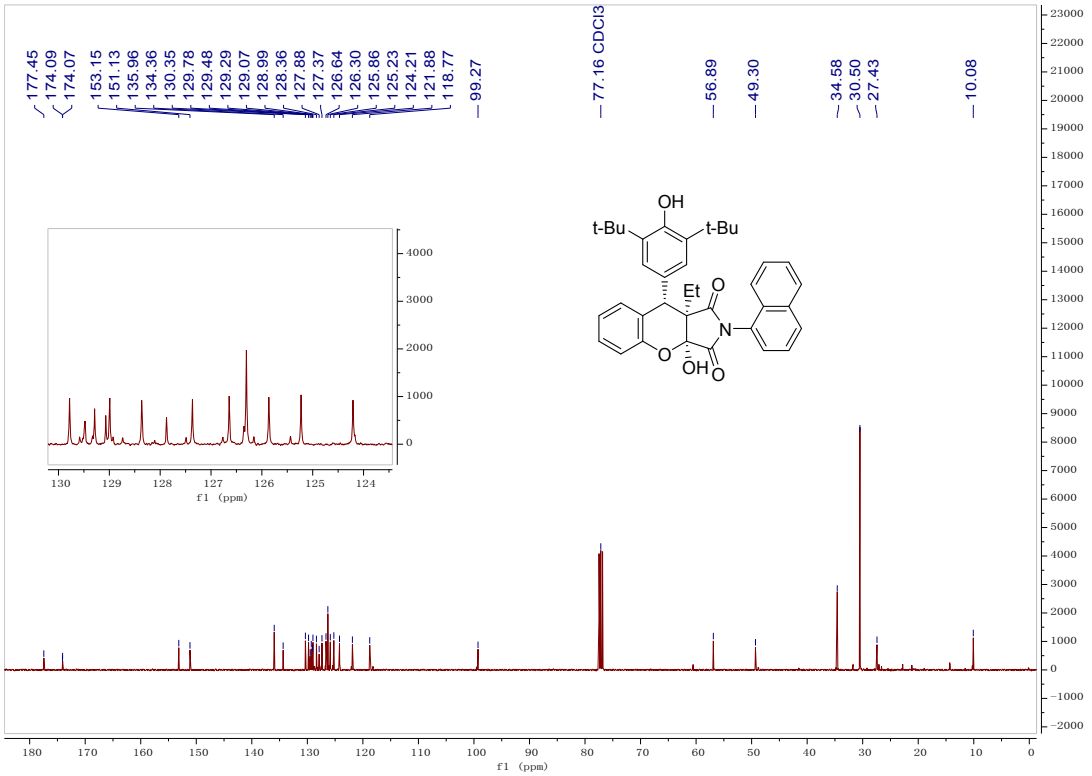
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 3.4 min, t_{major} = 3.8 min

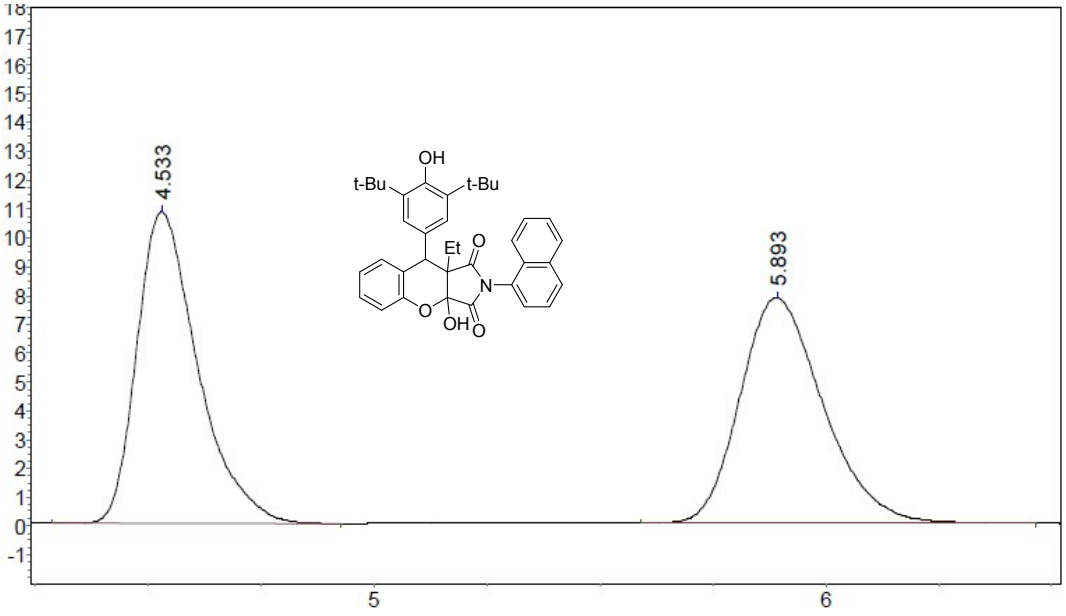
3na

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

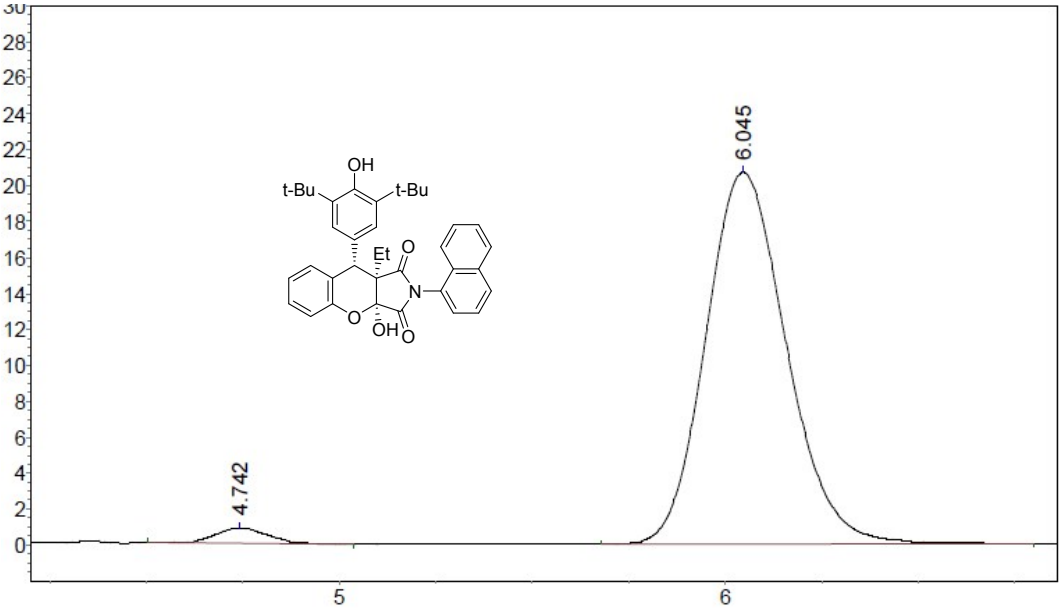


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.533	10811.977	101227.203	50.3186
2	5.893	7809.880	99945.297	49.6814

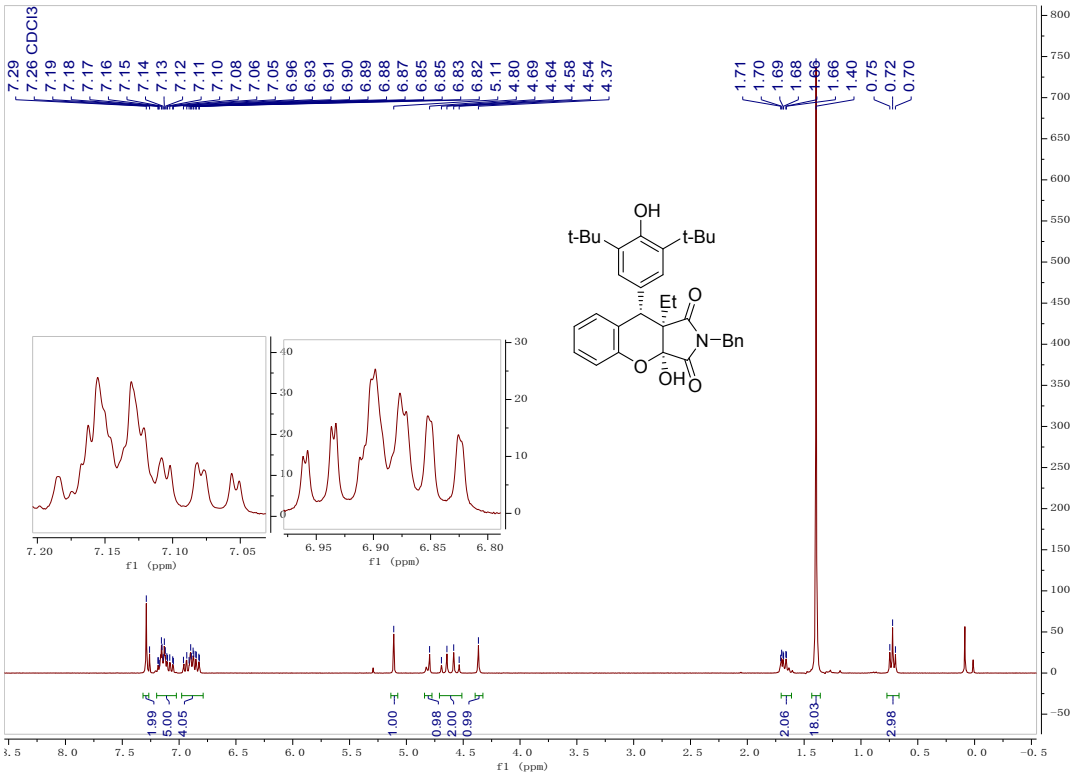


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.742	829.508	7541.141	2.4584
2	6.045	20658.809	299211.313	97.5416

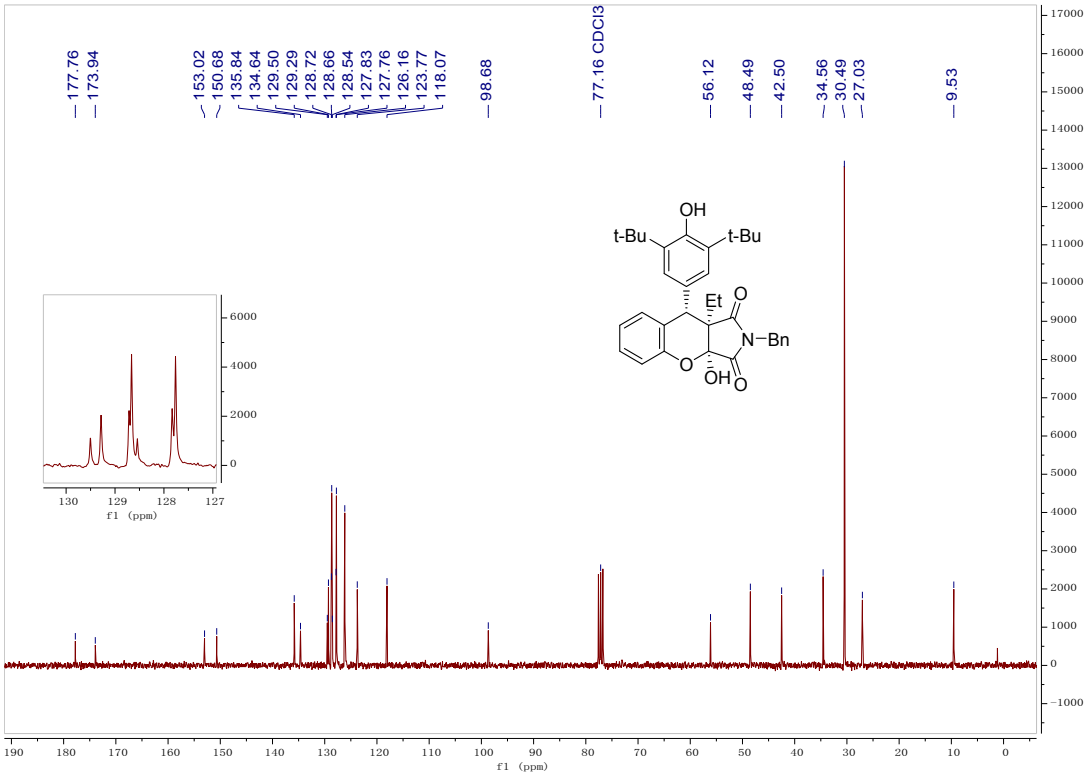
HPLC conditions: Chiralpak AY-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.7 min, t_{major} = 6.0 min.

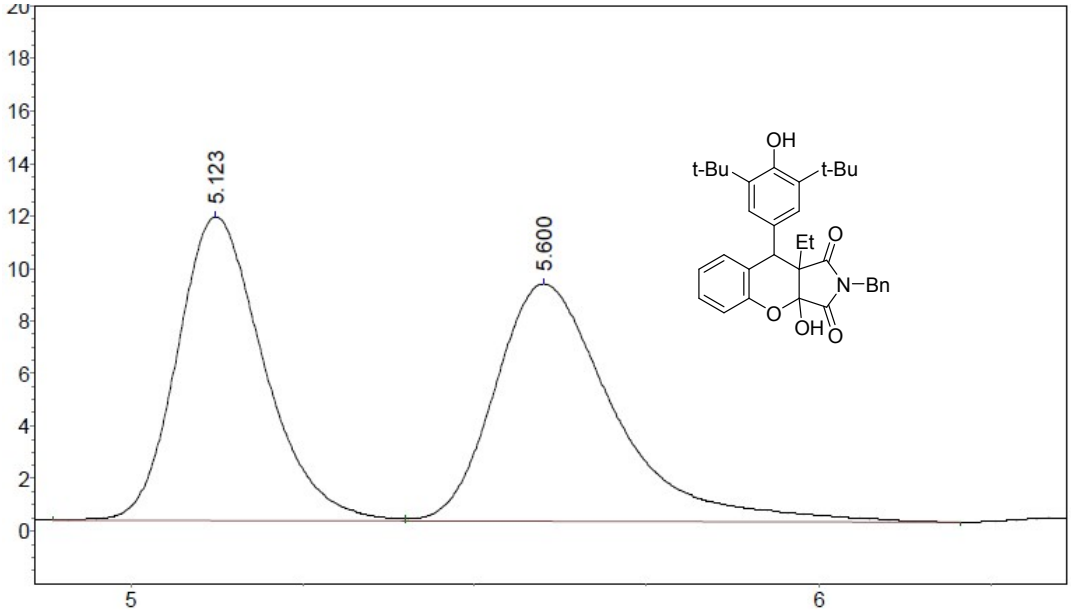
3oa

^{1}H NMR (300 MHz, $CDCl_3$)

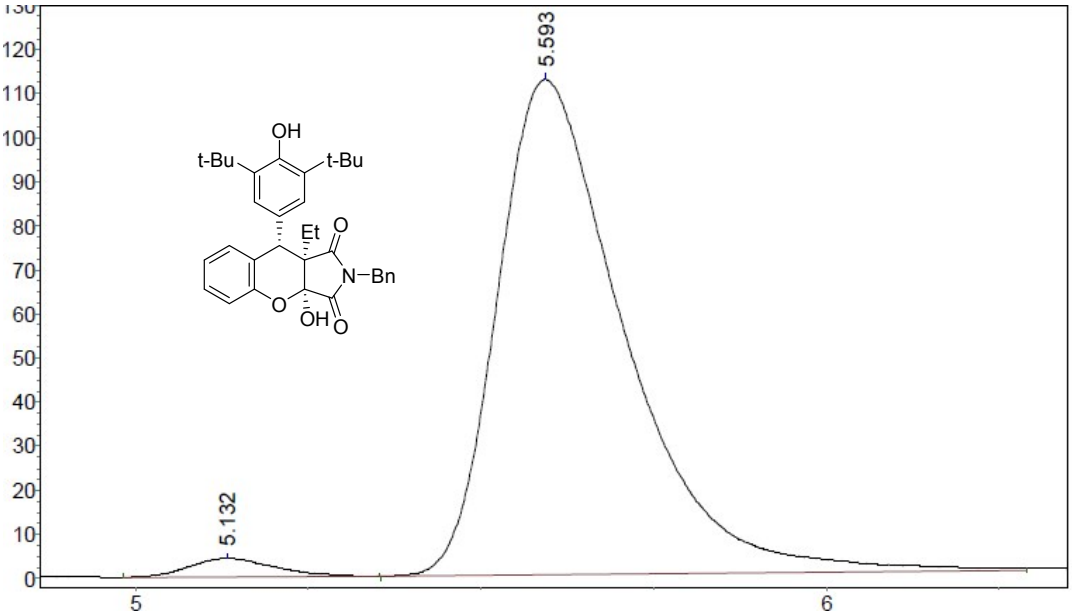


^{13}C NMR (300 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.123	11574.063	101221.063	48.2100
2	5.600	9036.477	108737.578	51.7900

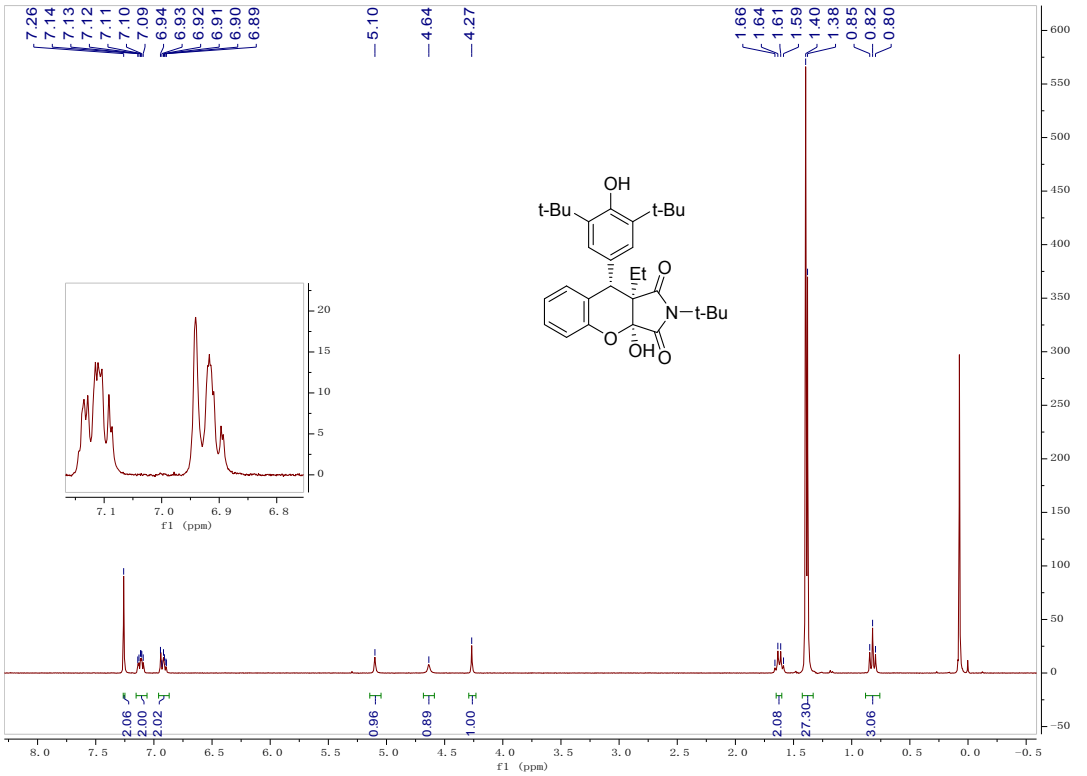


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.132	4173.631	36581.453	2.4754
2	5.593	112334.734	1441197.875	97.5246

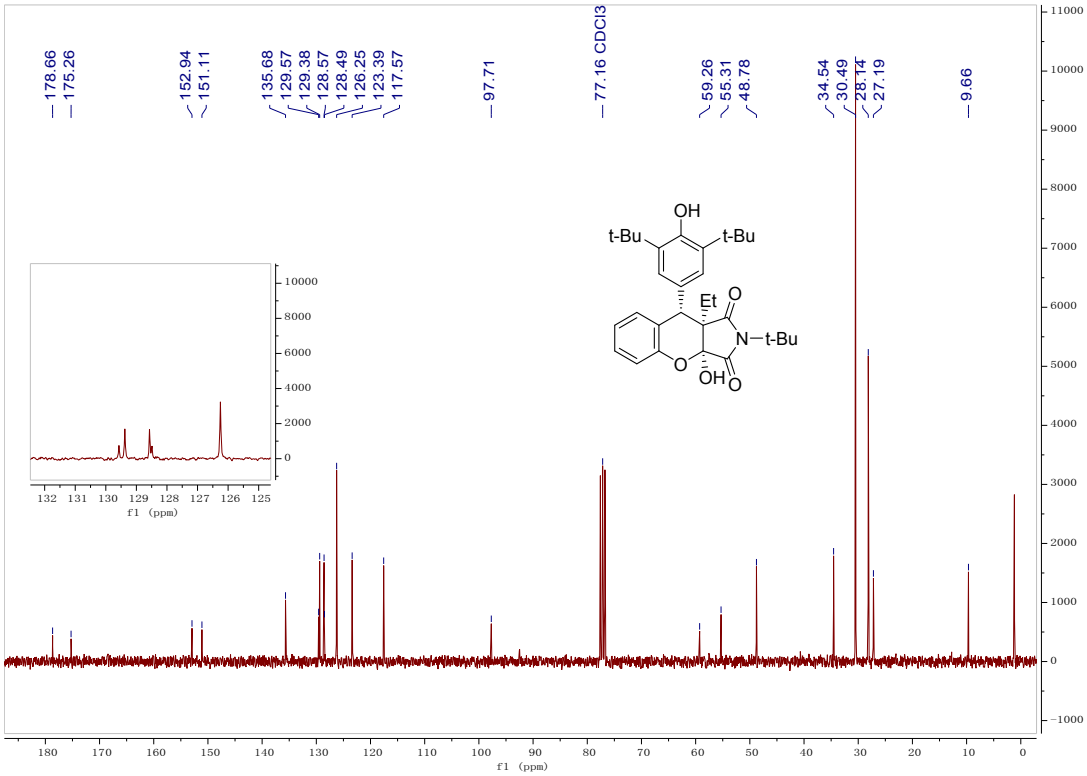
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 5.1 min, t_{major} = 5.5 min.

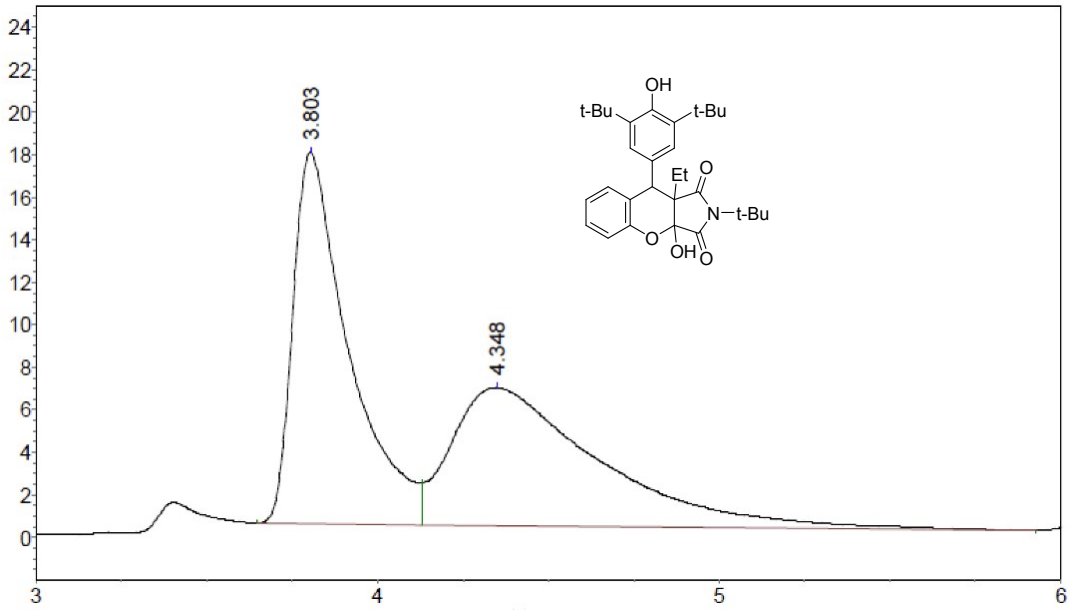
3pa

^{1}H NMR (300 MHz, $CDCl_3$)

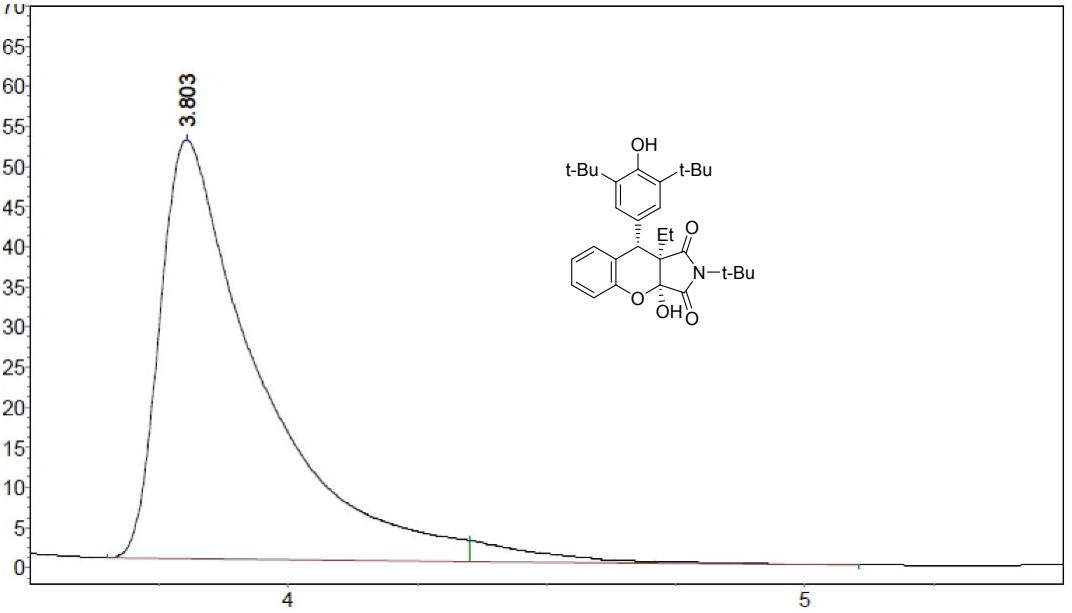


^{13}C NMR (75 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	3.803	17469.299	198530.953	50.4939
2	4.348	6471.702	194646.844	49.5061

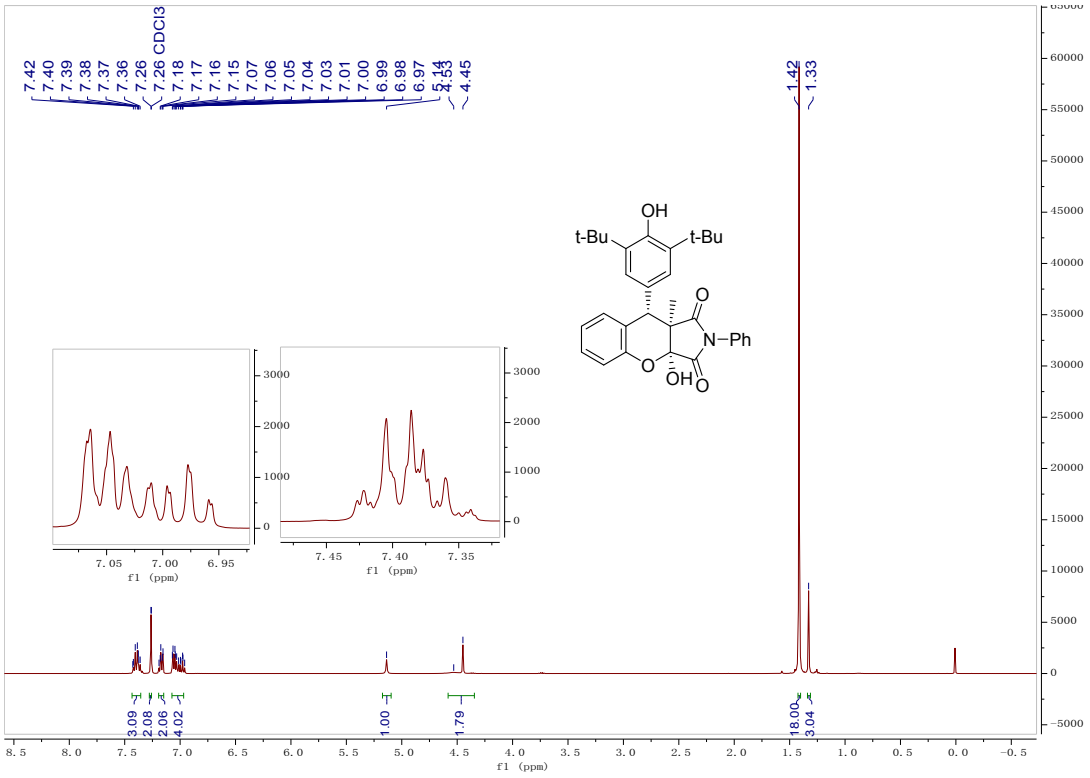


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	3.803	52225.031	698375.625	96.6858
2	4.345	2293.531	23938.984	3.3142

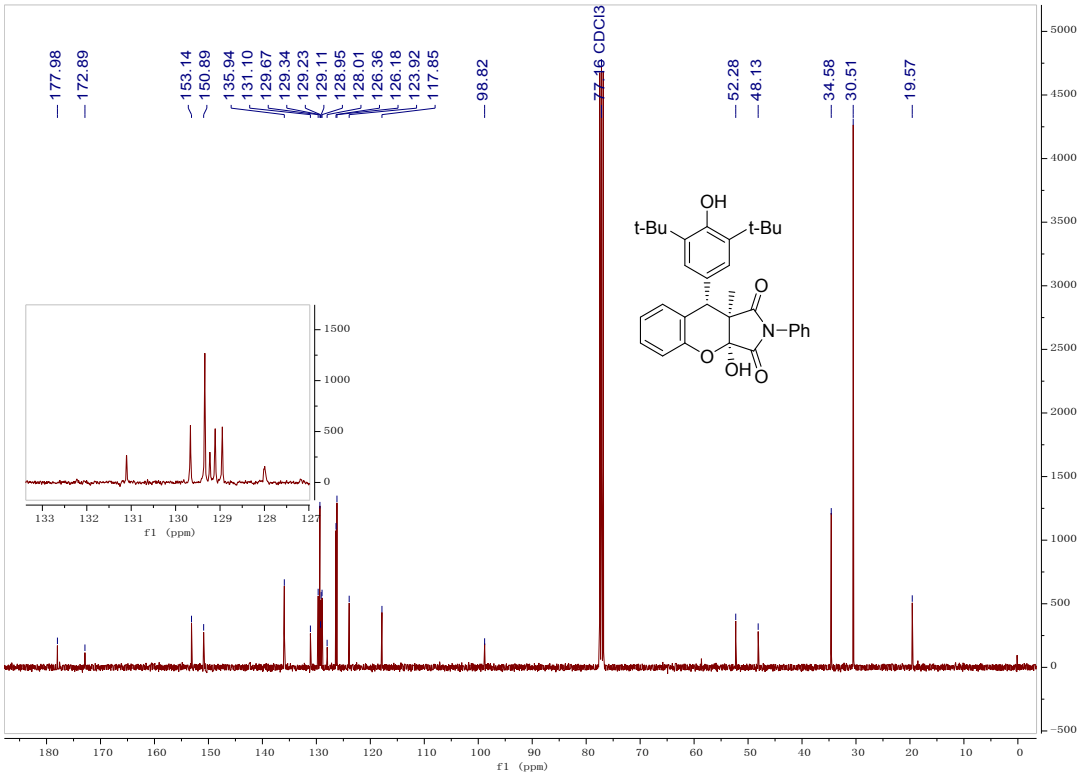
HPLC conditions: Chiralpak AY-H, *n*-hexane/isopropanol = 95/5, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 3.8 min, t_{major} = 4.3 min.

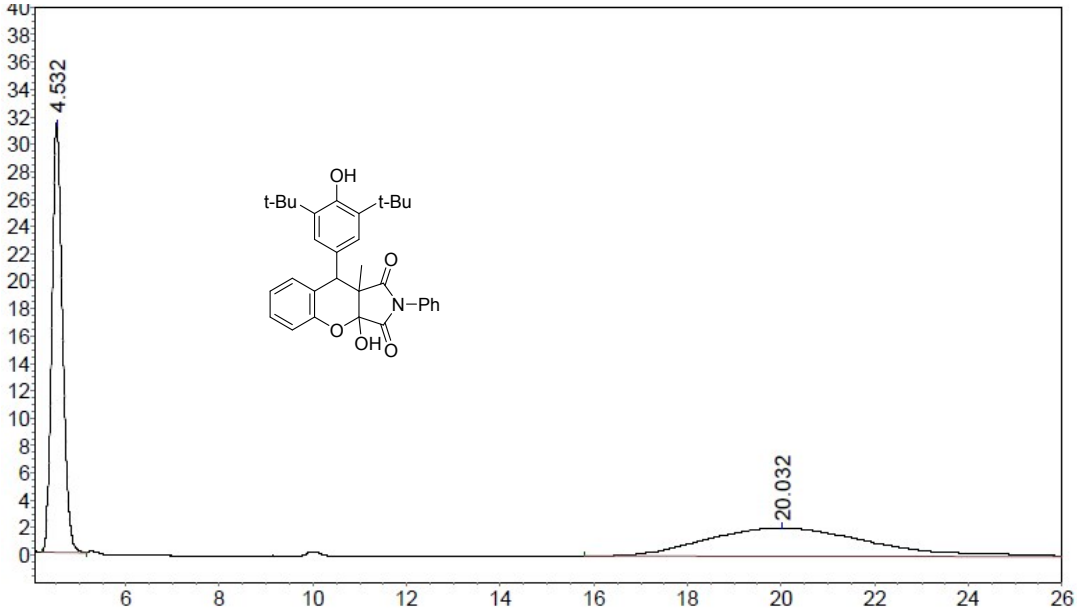
3qa

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

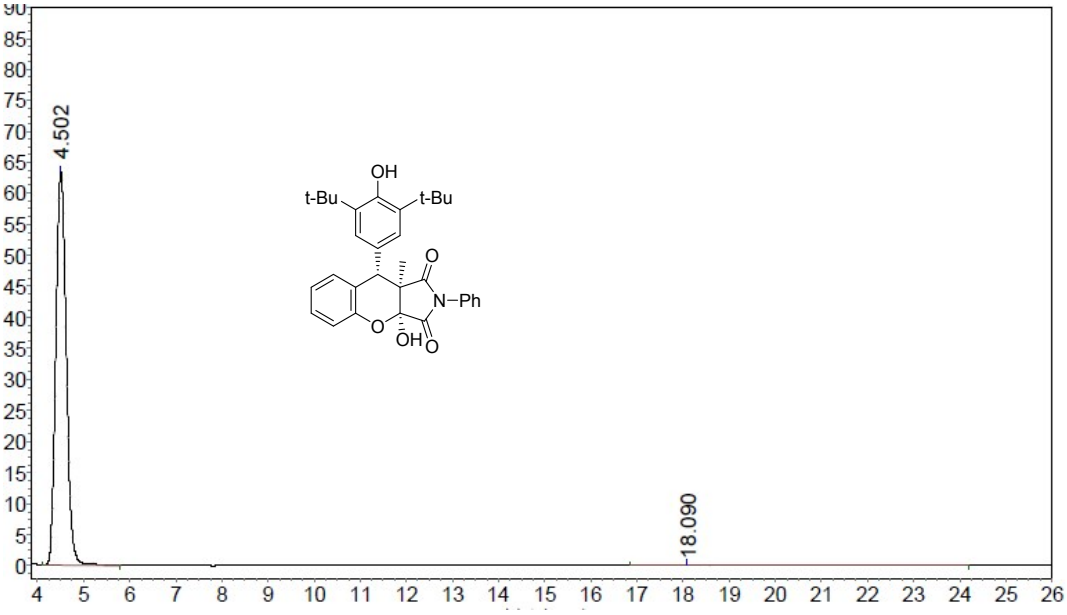


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.532	31145.725	501064.813	49.4508
2	20.032	2098.246	512194.594	50.5492

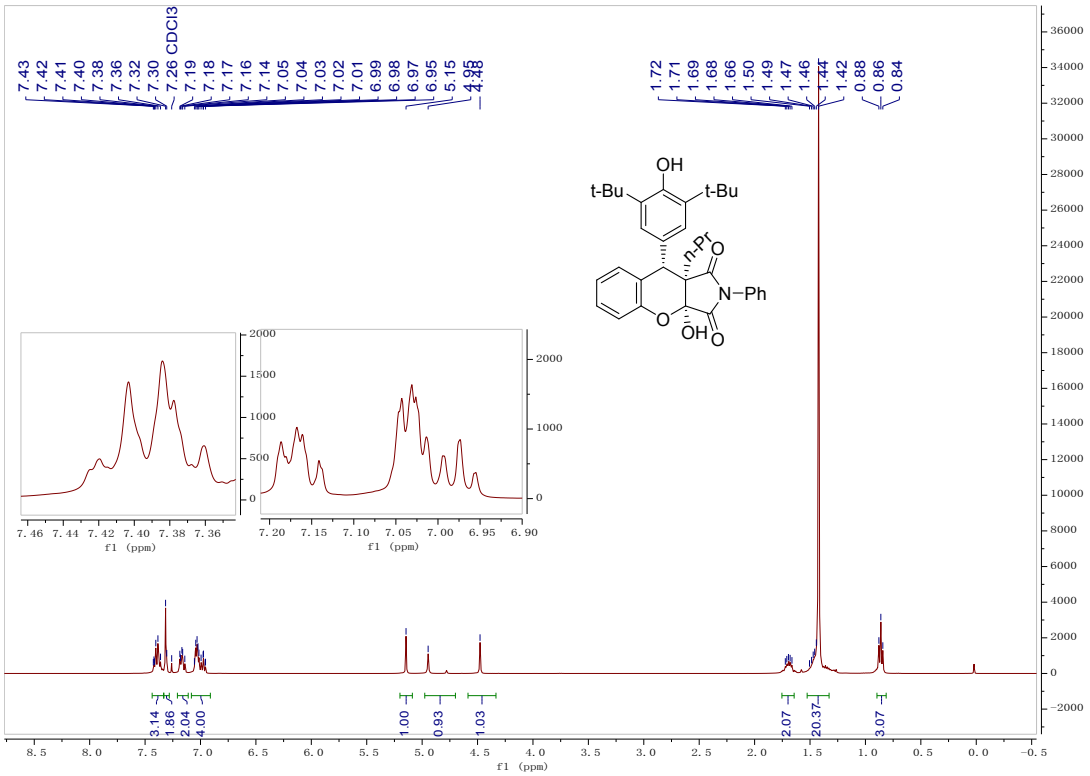


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.502	63386.730	983630.125	99.1920
2	18.090	74.707	8012.894	0.8080

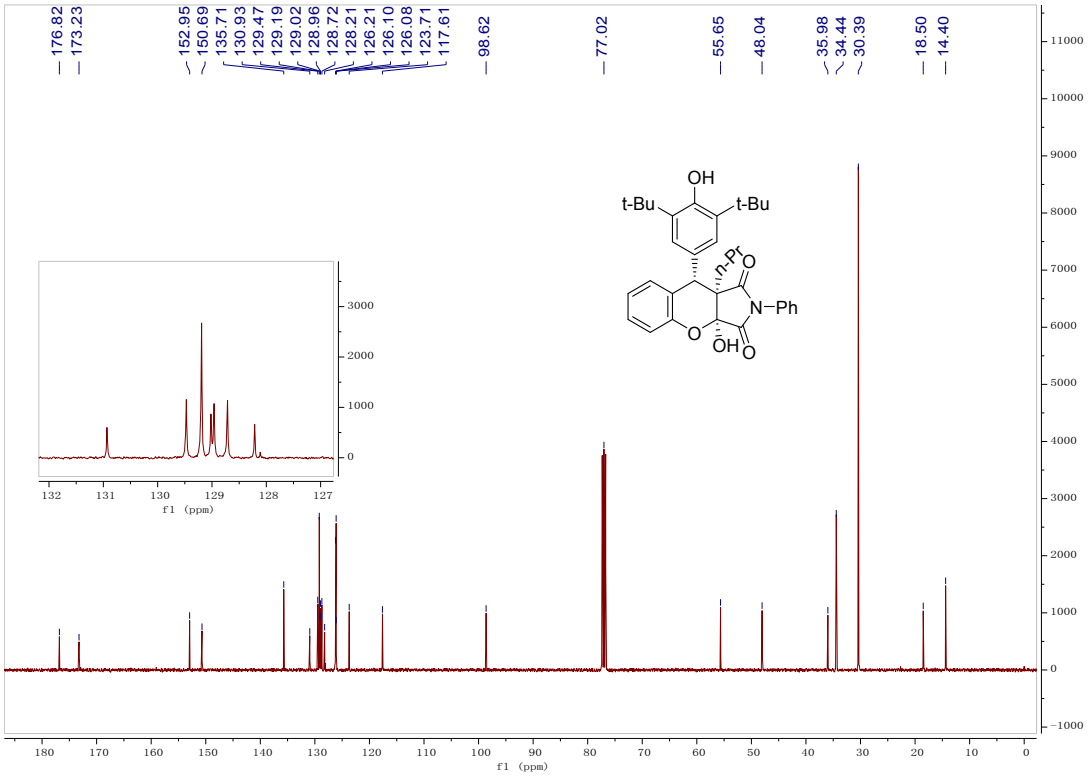
HPLC conditions: Chiralpak AS-H, *n*-hexane/isopropanol = 97/3, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.5 min, t_{major} = 18.0 min.

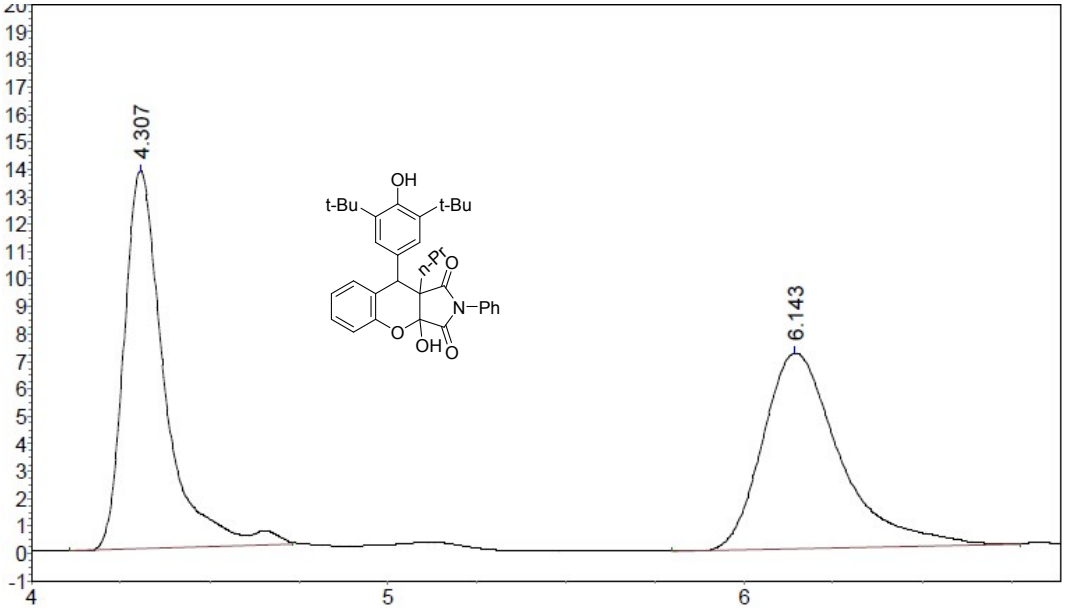
3ra

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

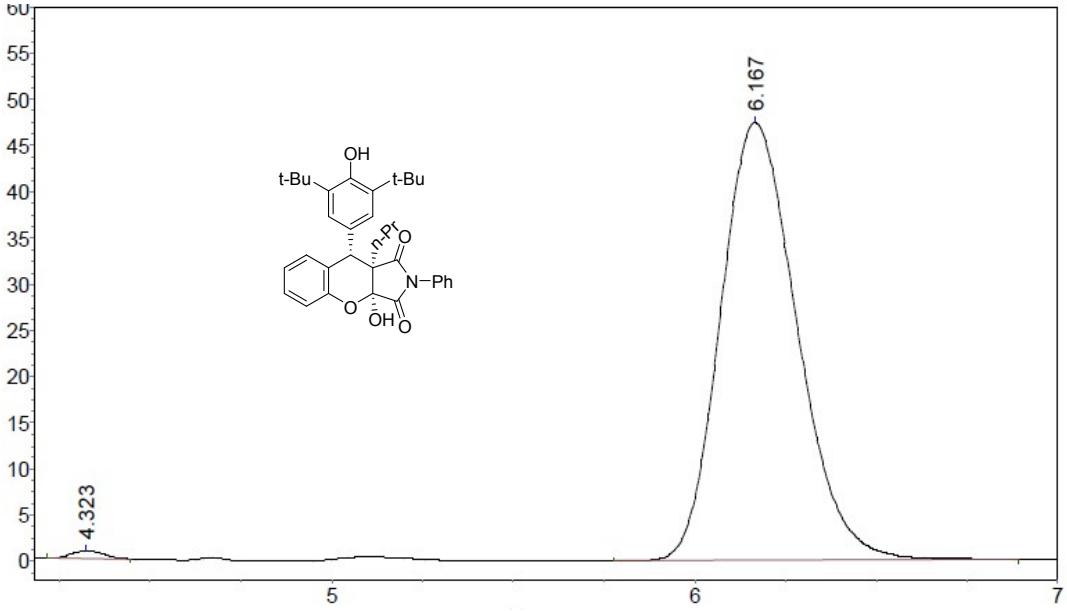


^{13}C NMR (100 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.307	13750.236	109111.117	50.3893
2	6.143	7138.573	107425.297	49.6107

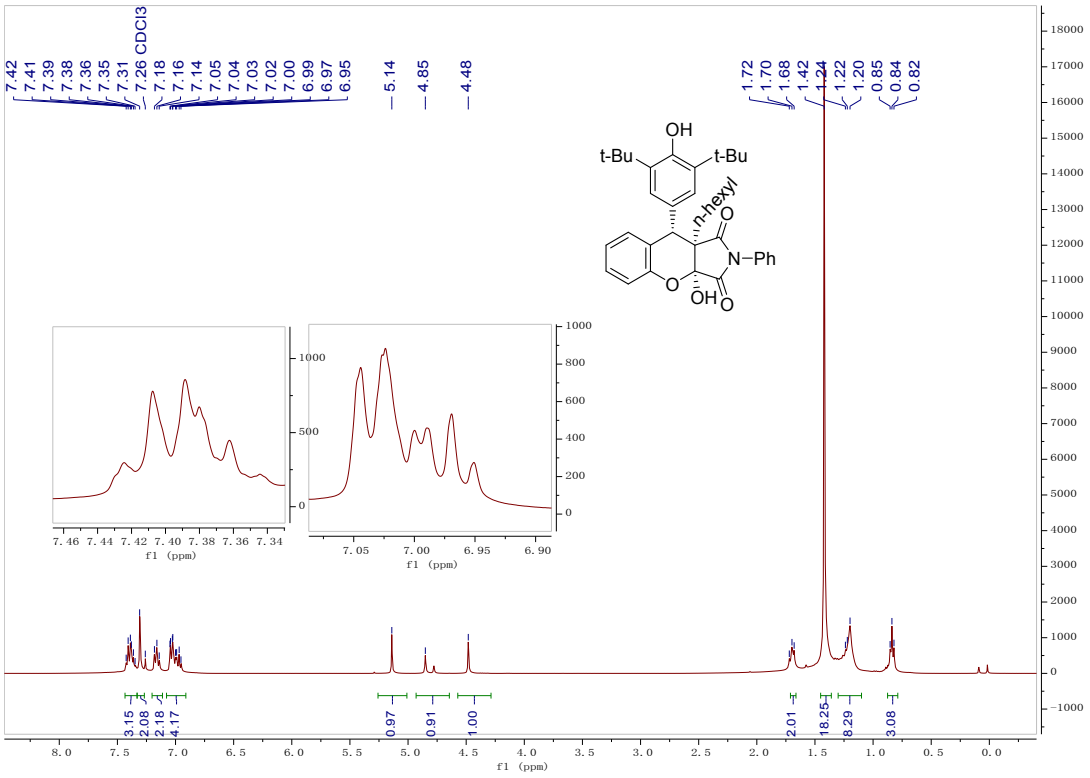


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.323	777.730	5173.700	0.7430
2	6.167	47335.461	691119.500	99.2570

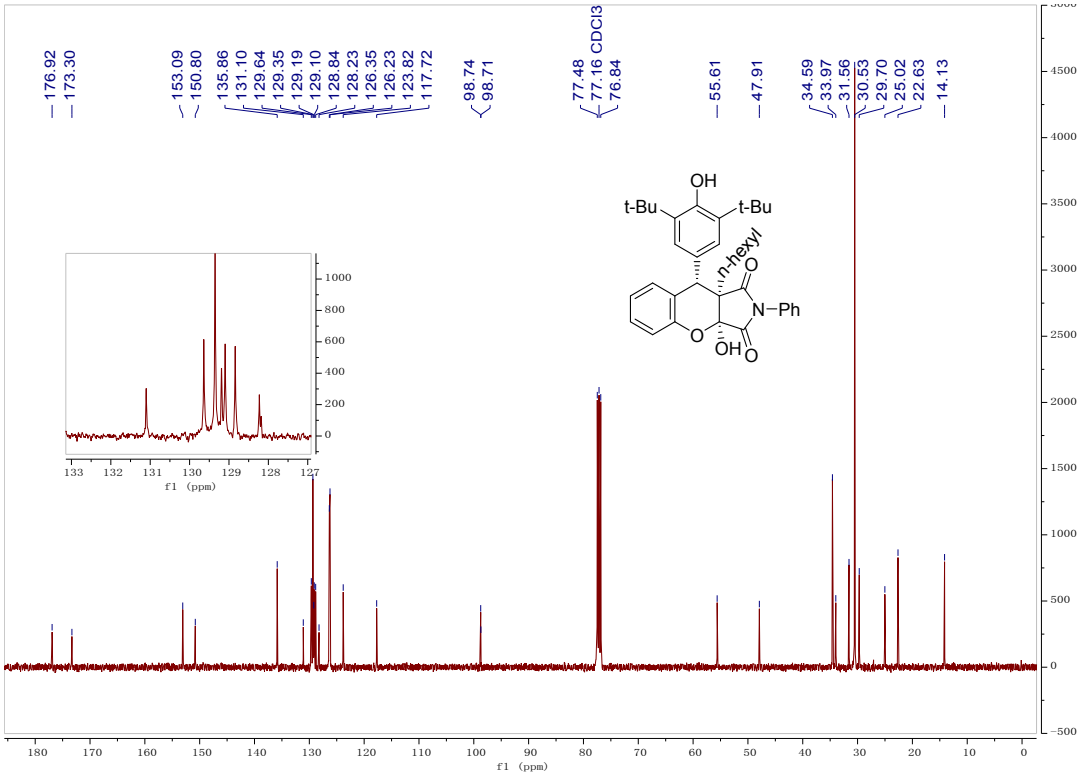
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.3 min, t_{major} = 6.1 min

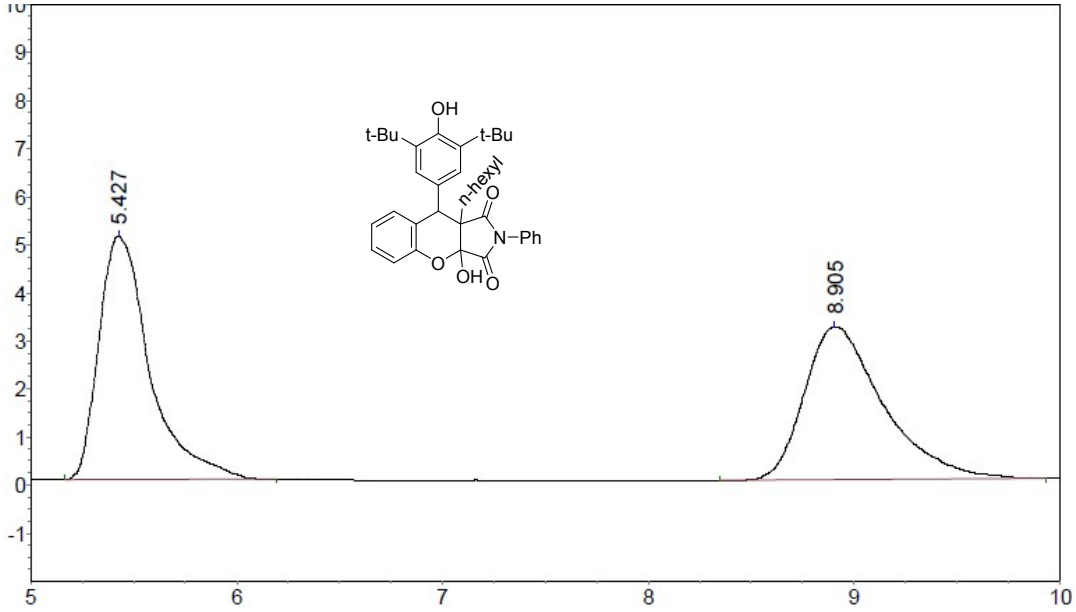
3sa

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

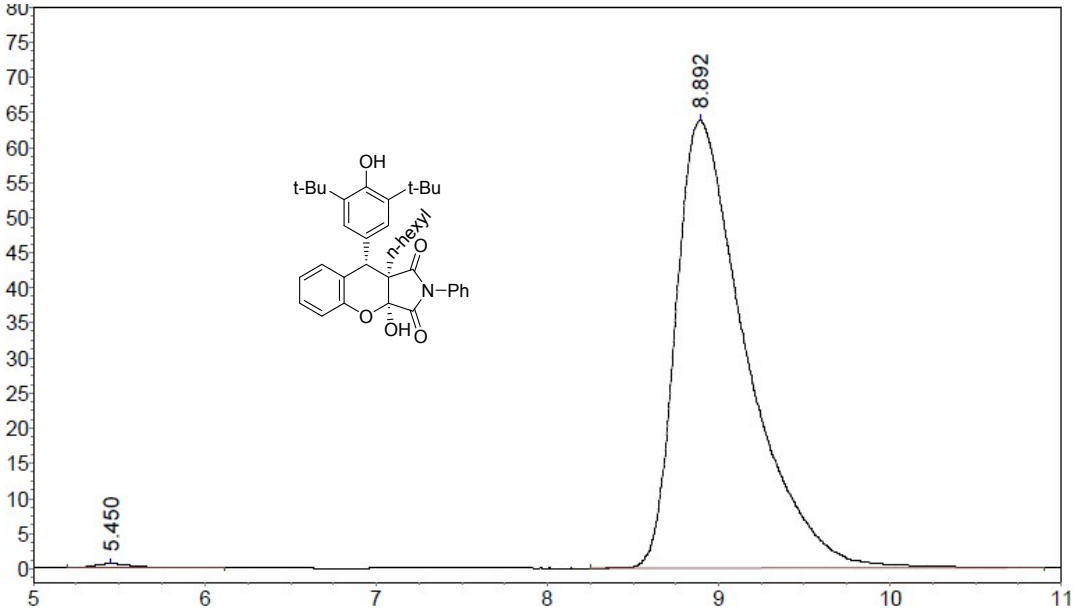


^{13}C NMR (400 MHz, $CDCl_3$)





Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.427	5057.725	86389.148	50.5700
2	8.905	3183.844	84441.500	49.4299

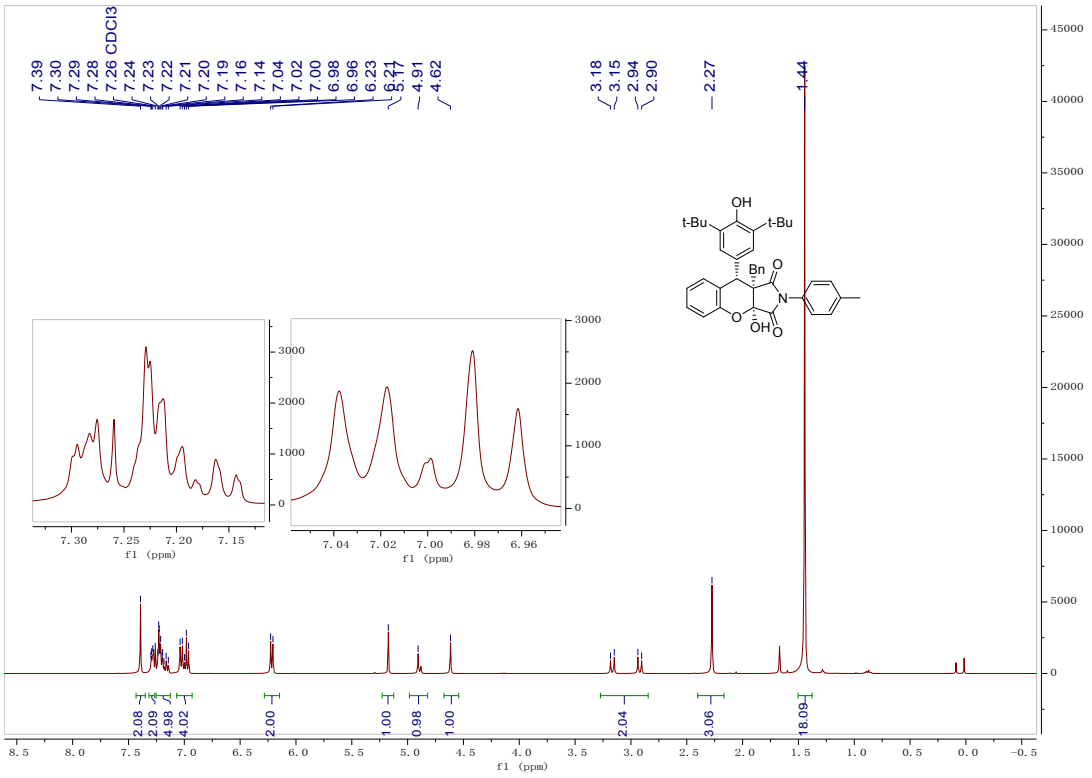


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.450	592.563	10247.850	0.5625
2	8.892	63768.621	1811448.250	99.4375

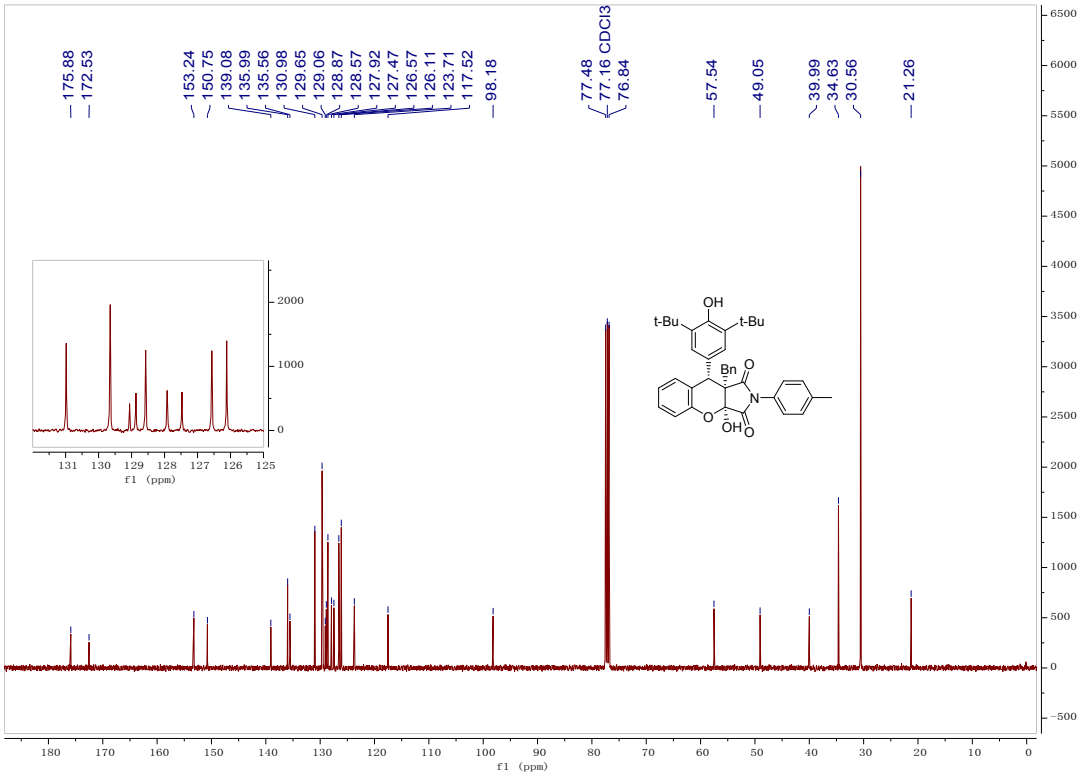
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 97/3, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 5.4 min, t_{major} = 8.8 min.

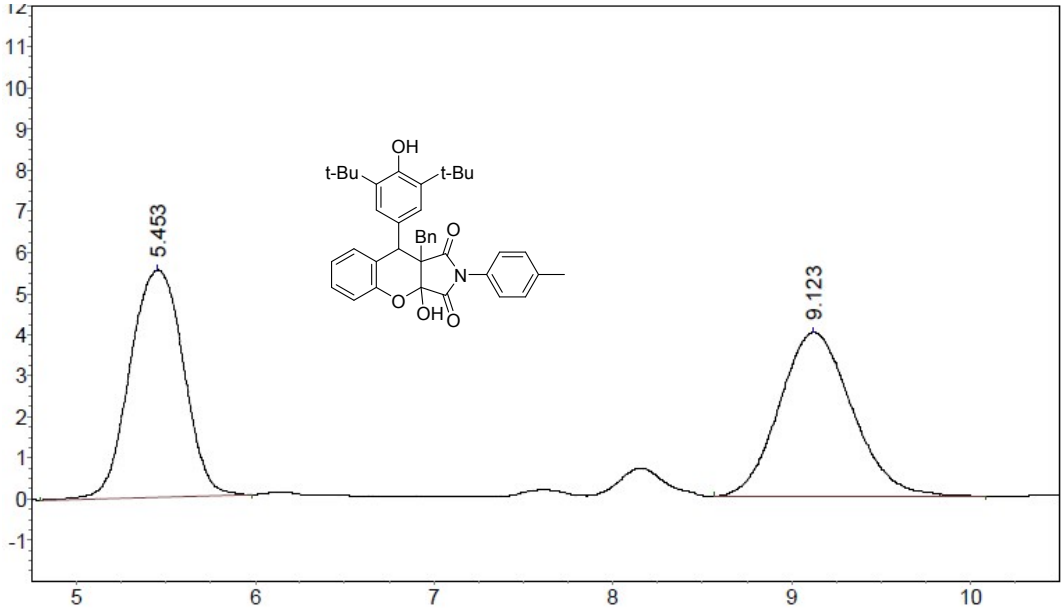
3ta

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

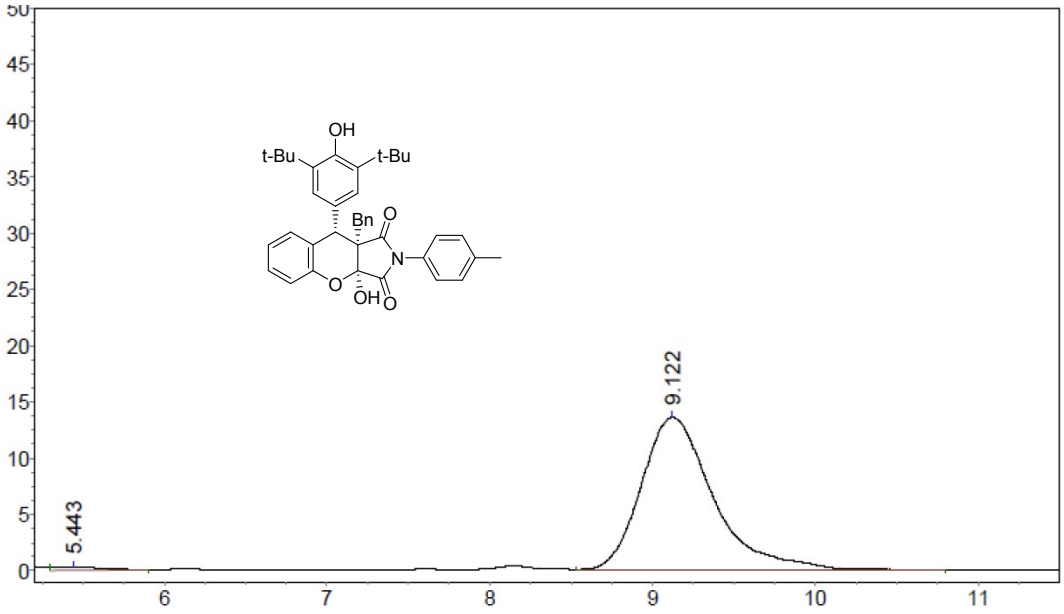


^{13}C NMR (100 MHz, $CDCl_3$)





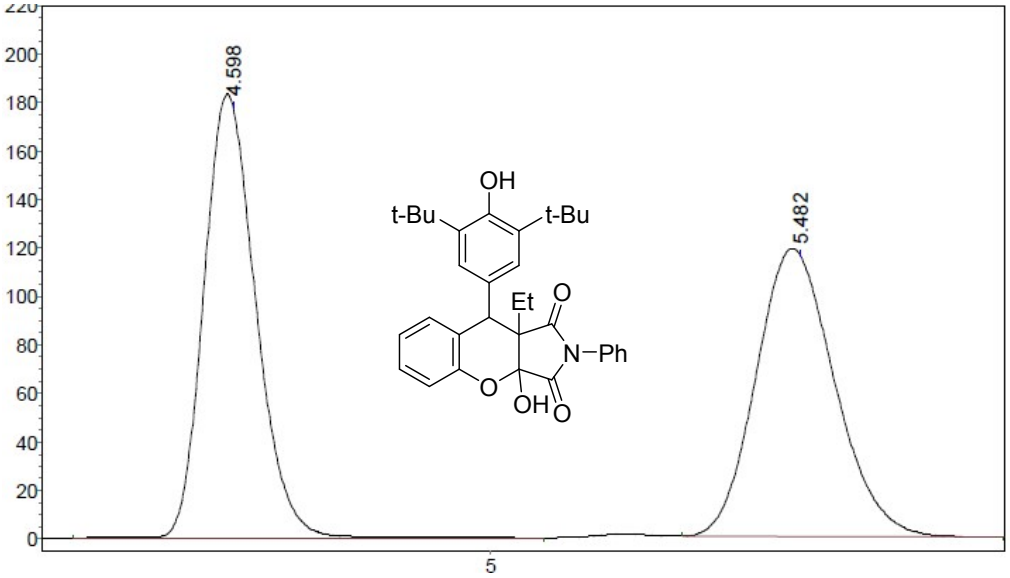
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.453	5512.300	114550.898	50.5632
2	9.123	3978.058	111999.000	49.4368



Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	5.443	319.831	4936.018	1.1631
2	9.122	13586.856	419461.250	98.8369

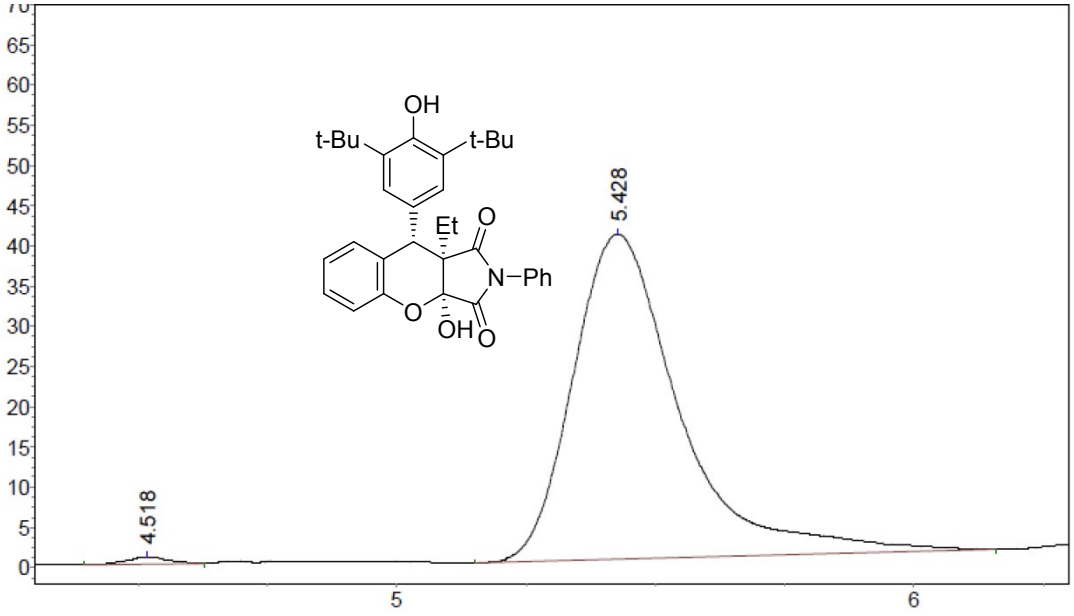
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 97/3, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 7.9 min, t_{major} = 9.1 min.

Scale-up:3aa, 3aaa



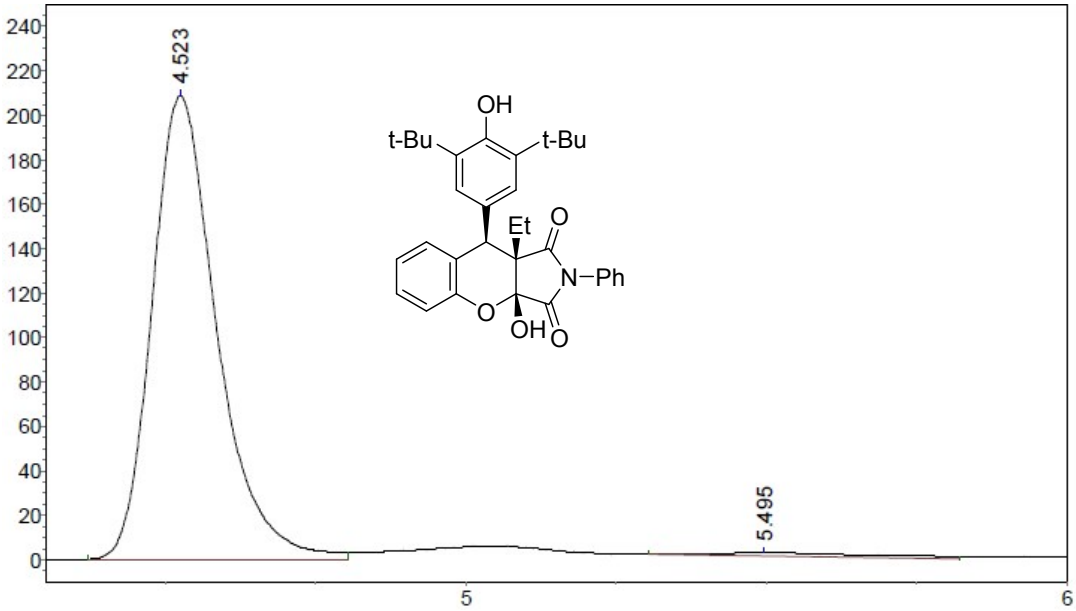
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.598	181363.234	1025639.500	50.1730
2	5.482	118320.695	1018568.438	49.8270

3aa



Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.518	799.000	4910.250	0.8419
2	5.428	40360.879	578320.000	99.1581

3aaa

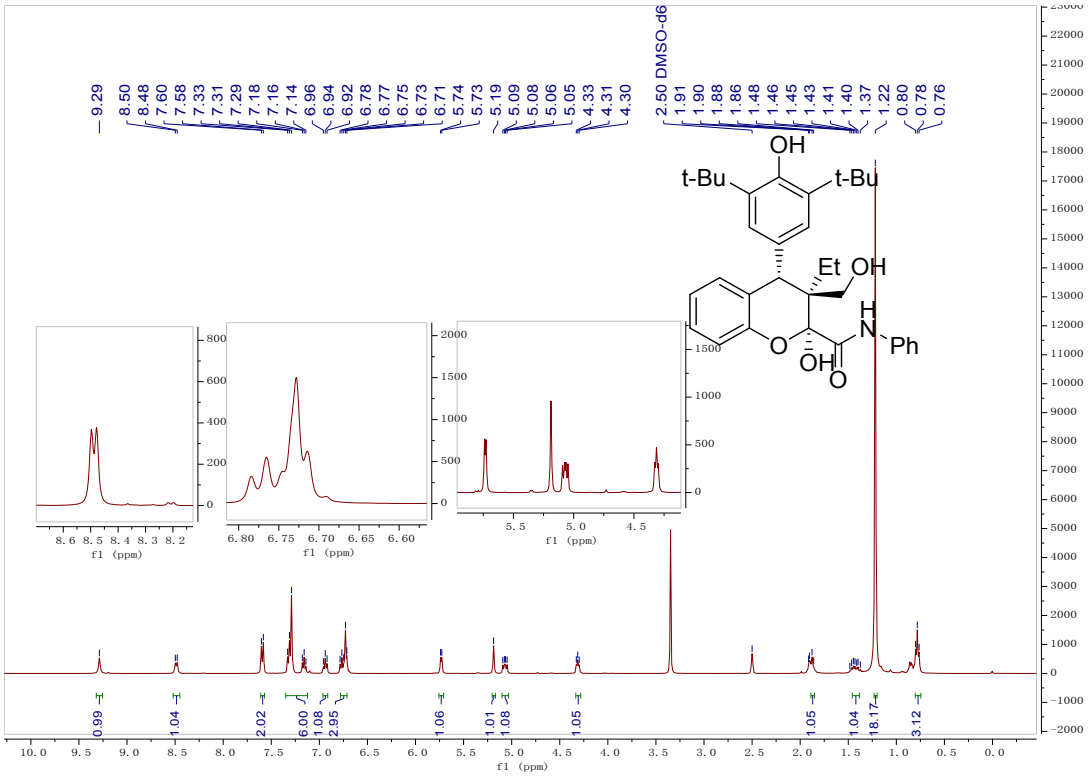


Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	4.523	208431.000	1558576.750	98.3563
2	5.495	1340.216	26046.600	1.6437

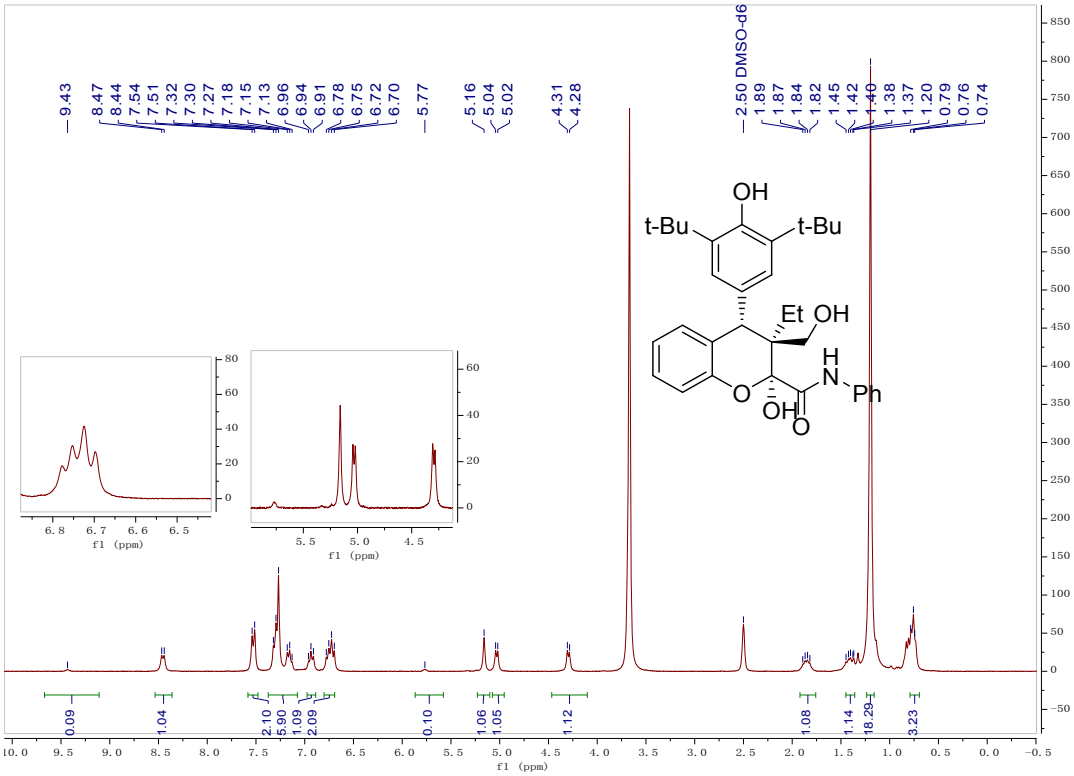
HPLC conditions: Chiralpak AD-H, *n*-hexane/isopropanol = 90/10, flow rate 1 mL/min, λ = 254 nm, t_{minor} = 4.5 min, t_{major} = 5.4 min.

6

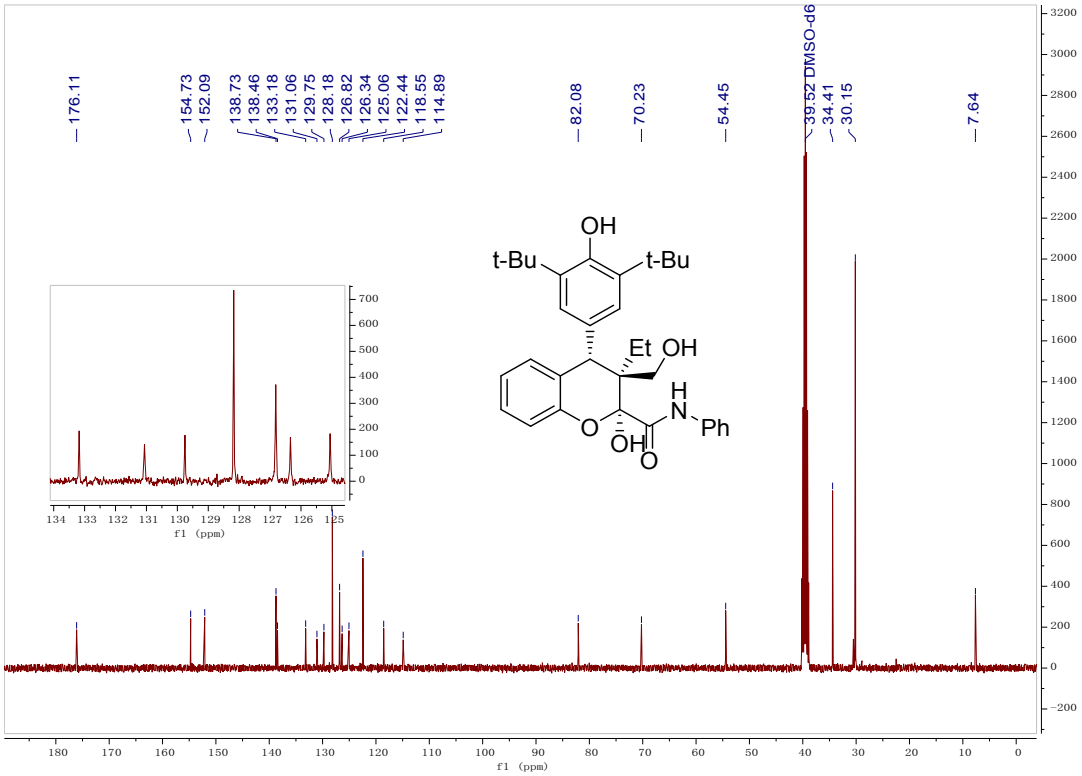
^{1}H NMR (400 MHz, DMSO-d_6)



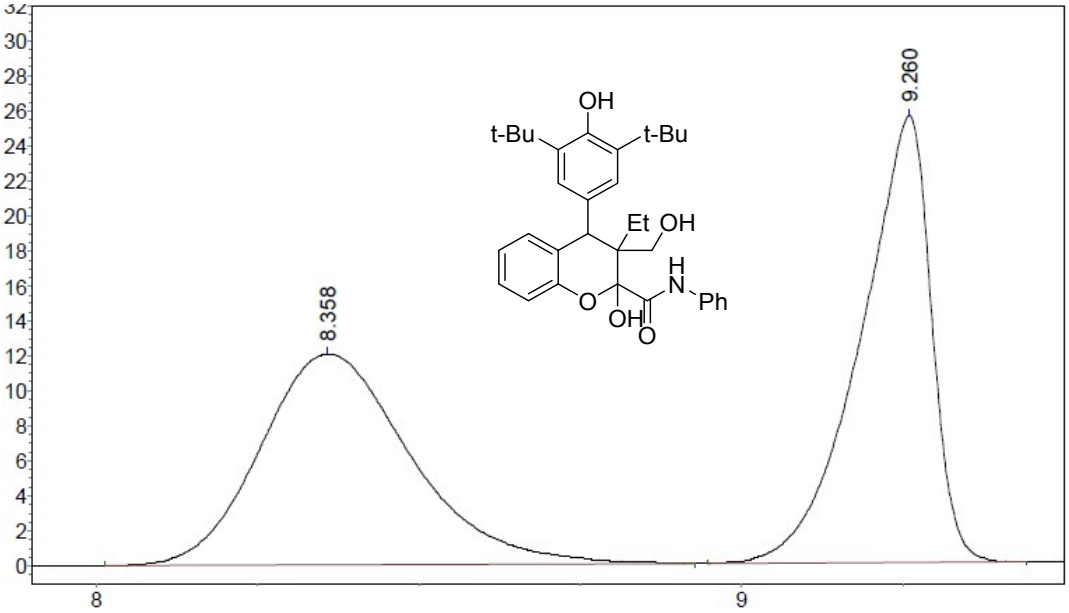
^{1}H NMR (300 MHz, DMSO-d_6, D_2O)



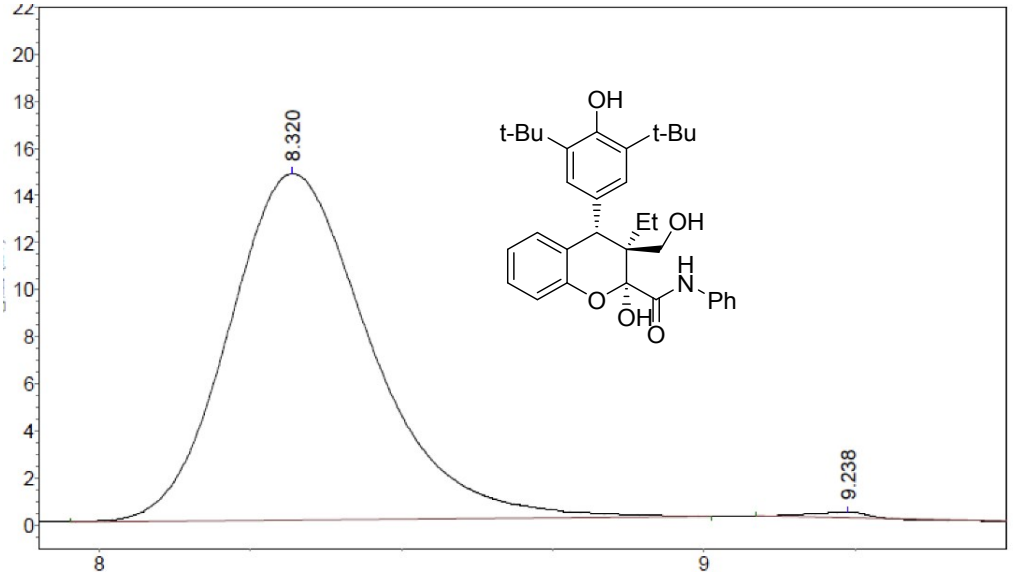
^{13}C NMR (400 MHz, DMSO-d_6)



6



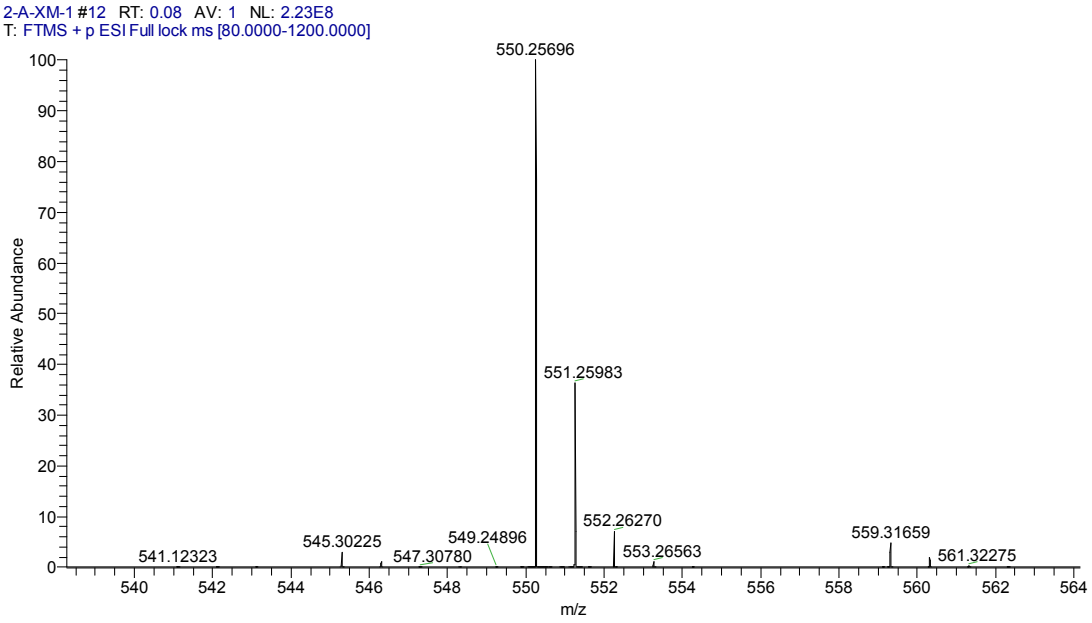
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	8.358	12064.592	194897.344	50.1558
2	9.260	25516.396	193686.250	49.8442



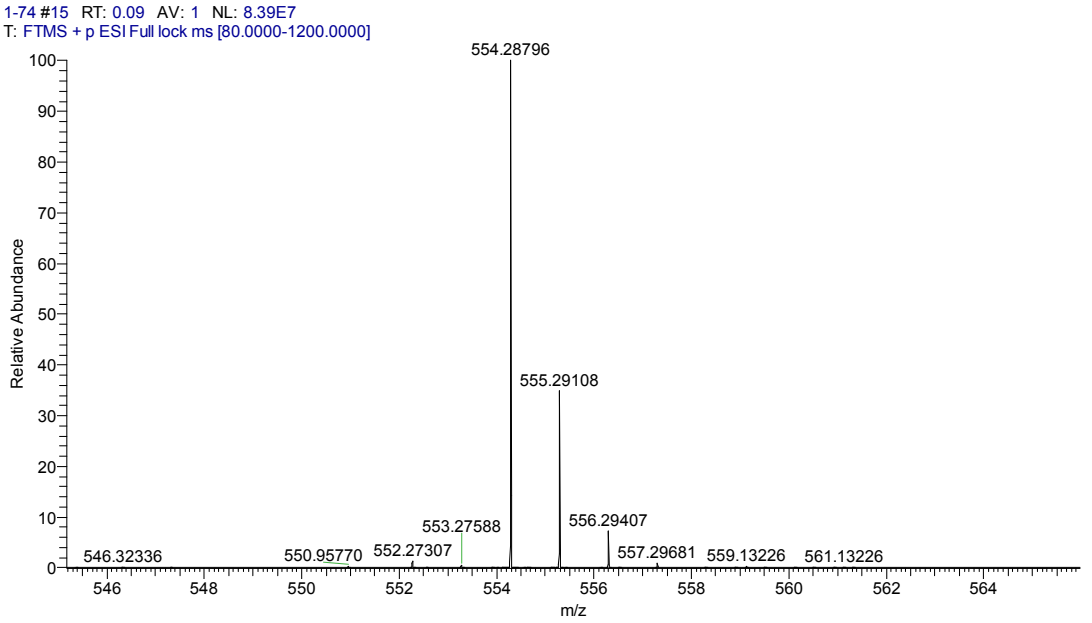
Peak#	Ret. Time (min)	Height (mV*sec)	Area (mv)	Area (%)
1	8.320	14713.543	239648.203	99.4408
2	9.238	253.302	1347.650	0.5592

HPLC conditions: Chiralpak IC, n-hexane/isopropanol = 97/3, flow rate 1 mL/min, λ = 254 nm, tminor = 8.3 min, tmajor = 9.2 min.

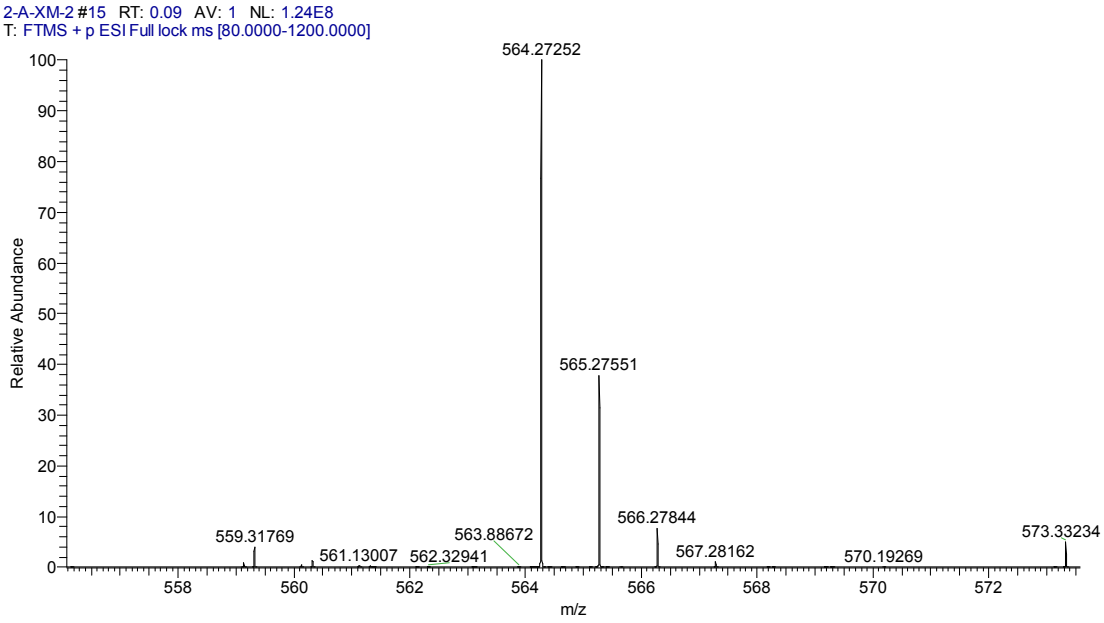
3aa


2-A-XM-1 #12 RT: 0.08 AV: 1 NL: 2.23E8
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
550.25696
551.25983
552.26270
553.26563
541.12323
545.30225
547.30780
549.24896
559.31659
561.32275
Relative Abundance
m/z

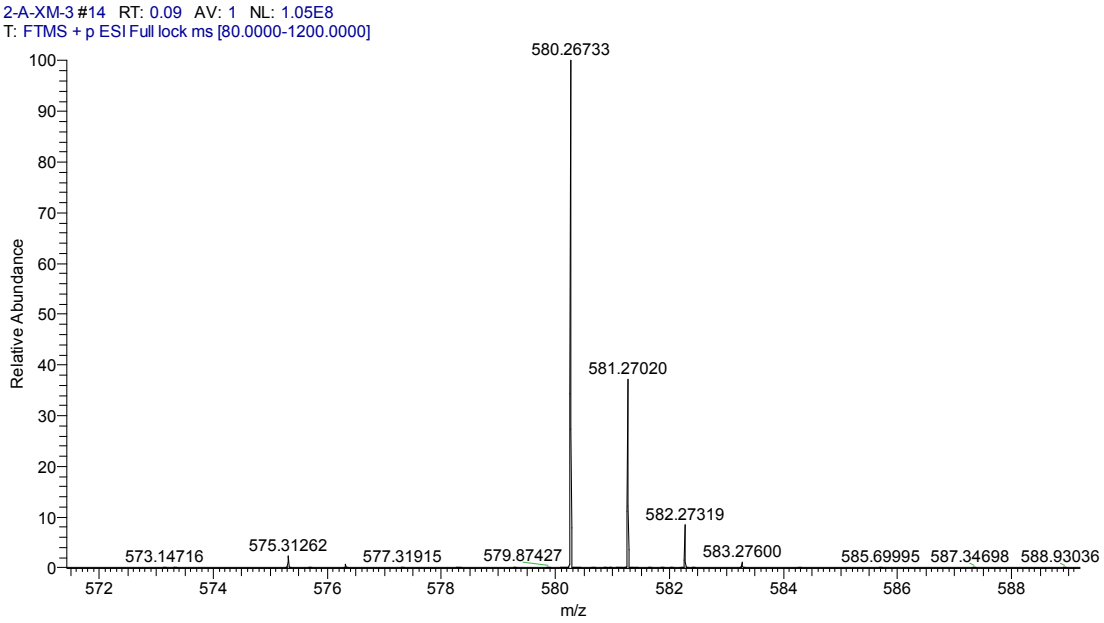
3aa'


1-74 #15 RT: 0.09 AV: 1 NL: 8.39E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
554.28796
555.29108
556.29407
557.29681
553.27588
552.27307
550.95770
546.32336
559.13226
561.13226
Relative Abundance
m/z

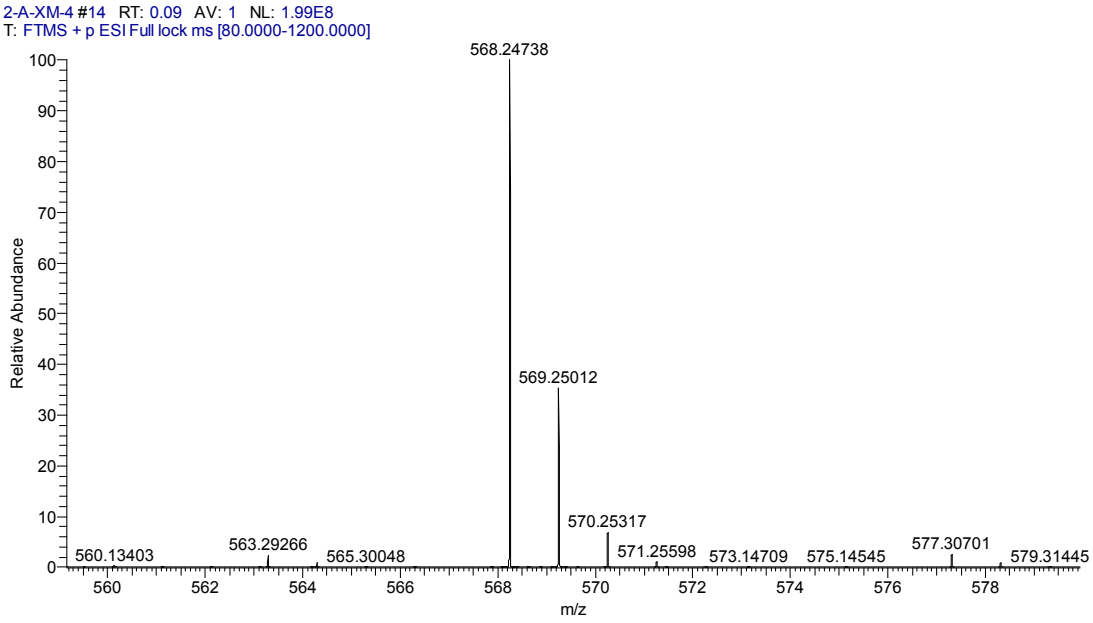
3ab


2-A-XM-2 #15 RT: 0.09 AV: 1 NL: 1.24E8
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
564.27252
565.27551
566.27844
559.31769
563.88672
573.33234
561.13007
562.32941
567.28162
570.19269
Relative Abundance
m/z

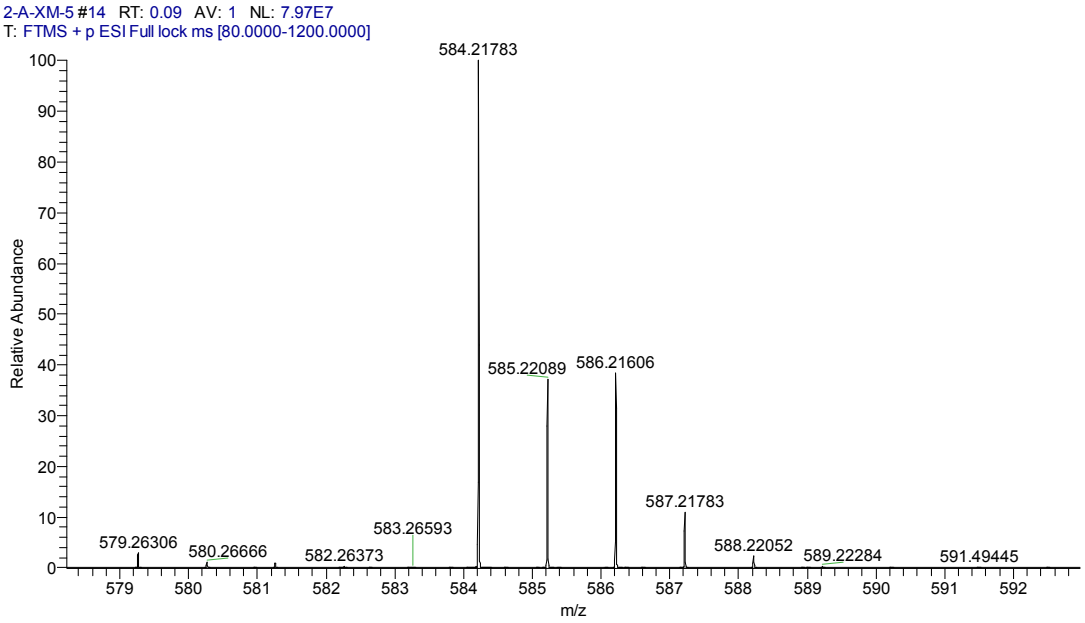
3ac


2-A-XM-3 #14 RT: 0.09 AV: 1 NL: 1.05E8
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
580.26733
581.27020
582.27319
573.14716
575.31262
577.31915
579.87427
583.27600
585.69995
587.34698
588.93036
Relative Abundance
m/z

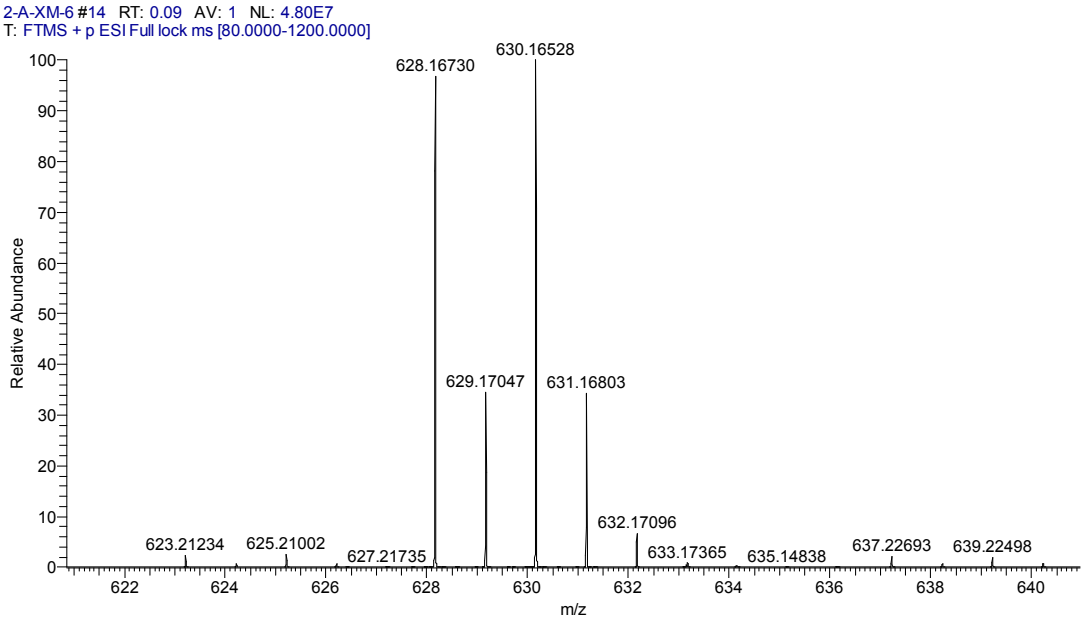
3ad


2-A-XM-4 #14 RT: 0.09 AV: 1 NL: 1.99E8
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
568.24738
569.25012
570.25317
560.13403
563.29266
565.30048
571.25598
573.14709
575.14545
577.30701
579.31445
Relative Abundance
m/z

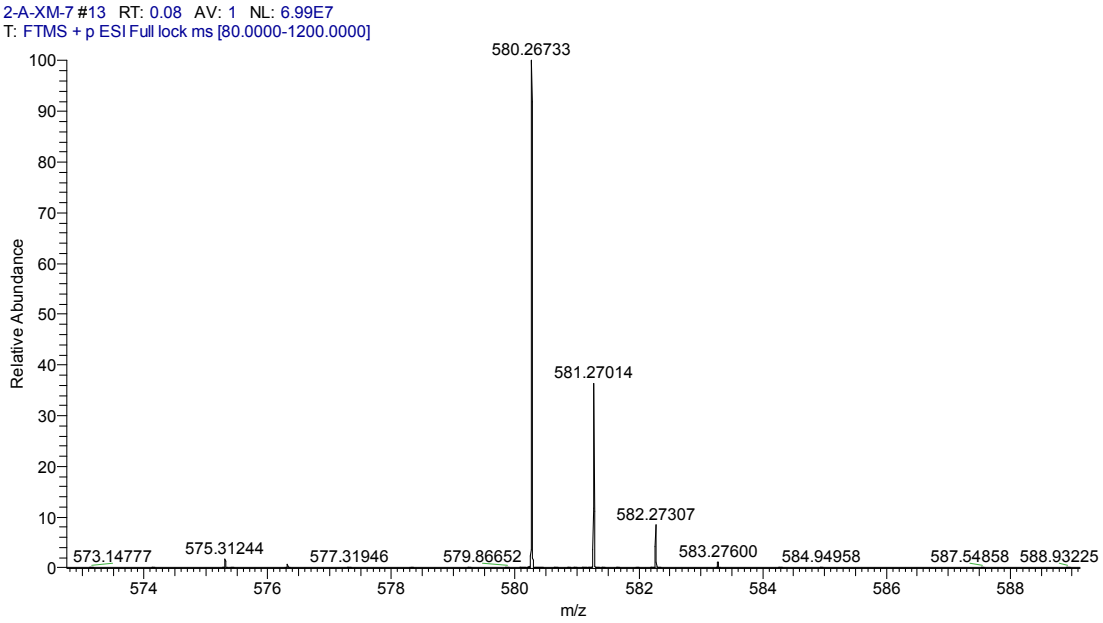
3ae


2-A-XM-5 #14 RT: 0.09 AV: 1 NL: 7.97E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
584.21783
585.22089
586.21606
587.21783
579.26306
580.26666
582.26373
583.26593
588.22052
589.22284
591.49445
Relative Abundance
m/z

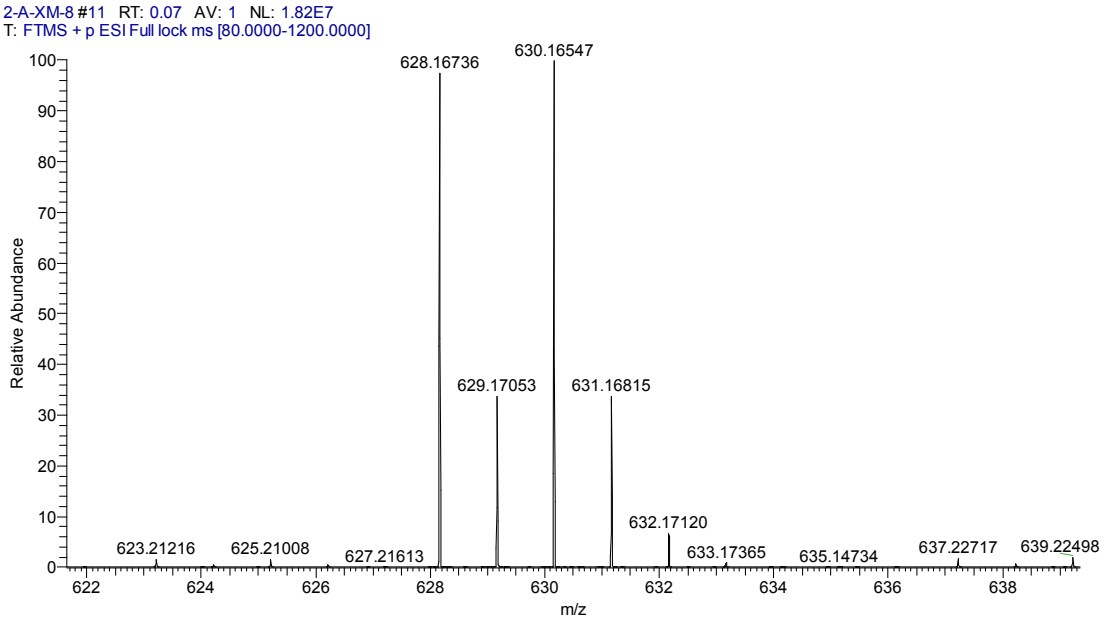
3af


2-A-XM-6 #14 RT: 0.09 AV: 1 NL: 4.80E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
628.16730
630.16528
629.17047
631.16803
632.17096
623.21234
625.21002
627.21735
633.17365
635.14838
637.22693
639.22498
Relative Abundance
m/z

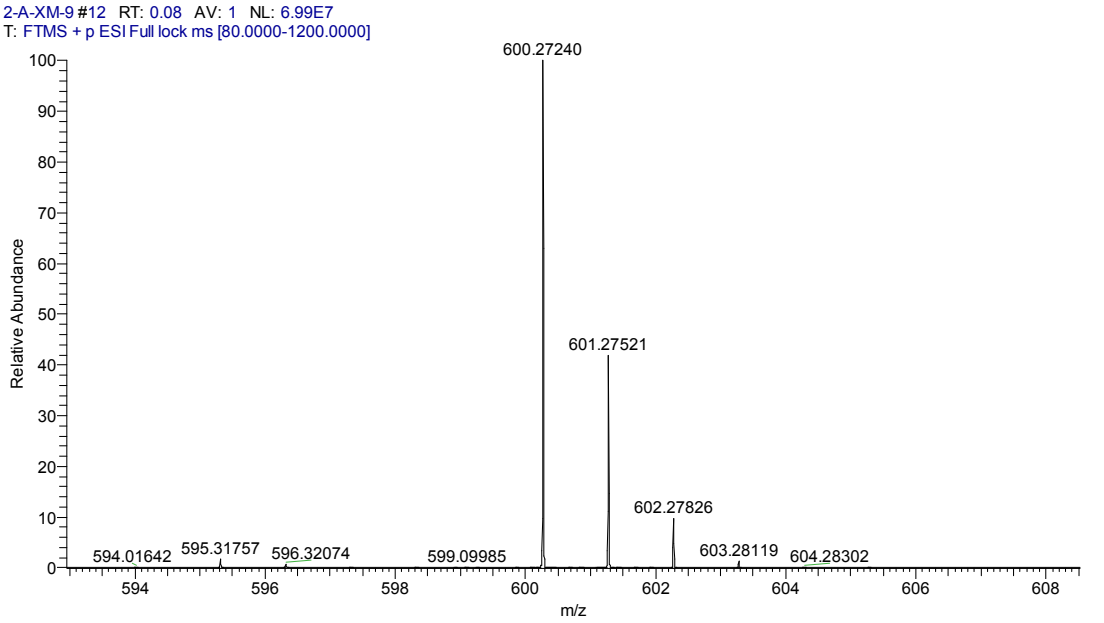
3ag


2-A-XM-7 #13 RT: 0.08 AV: 1 NL: 6.99E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
580.26733
581.27014
582.27307
573.14777
575.31244
577.31946
579.86652
583.27600
584.94958
587.54858
588.93225
Relative Abundance
m/z

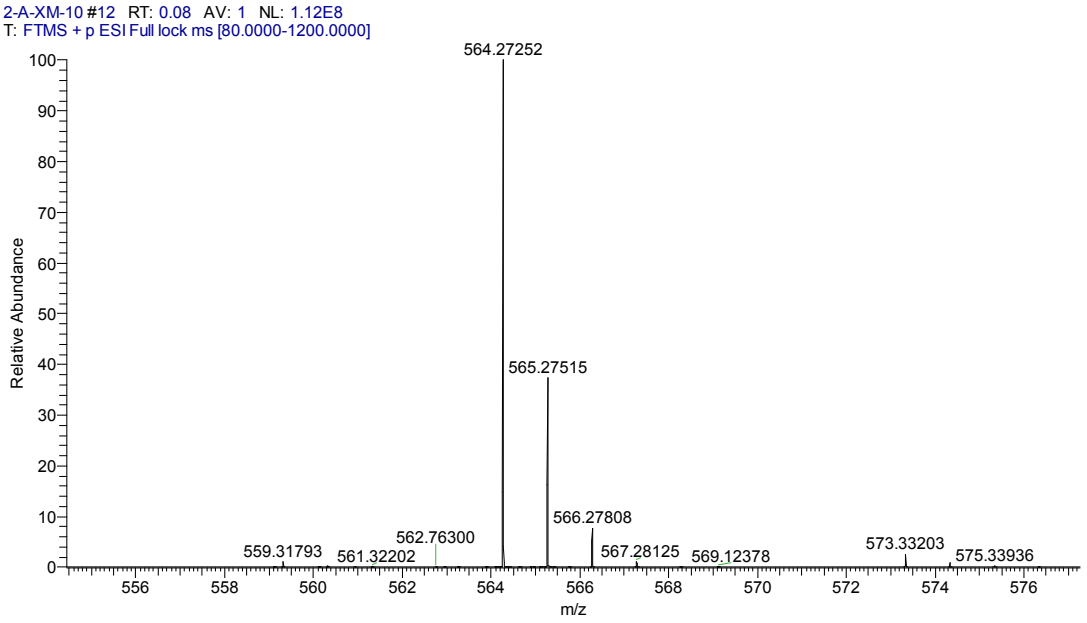
3ah


2-A-XM-8 #11 RT: 0.07 AV: 1 NL: 1.82E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
628.16736
630.16547
629.17053
631.16815
632.17120
623.21216
625.21008
627.21613
633.17365
635.14734
637.22717
639.22498
Relative Abundance
m/z

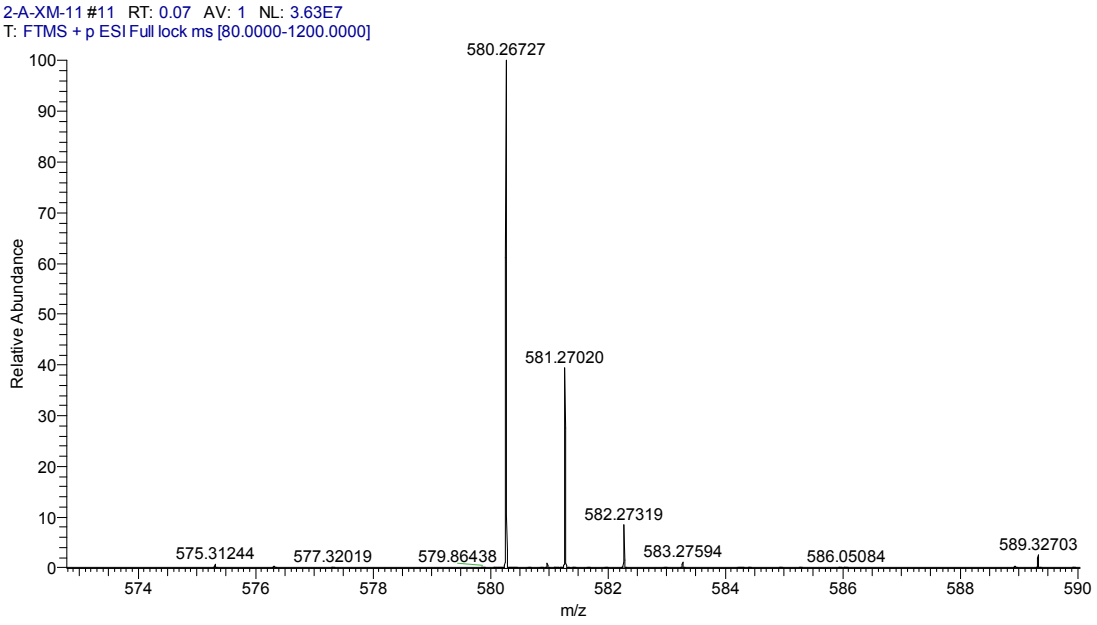
3ai


2-A-XM-9 #12 RT: 0.08 AV: 1 NL: 6.99E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
600.27240
601.27521
602.27826
594.01642
595.31757
596.32074
599.09985
603.28119
604.28302
Relative Abundance
m/z

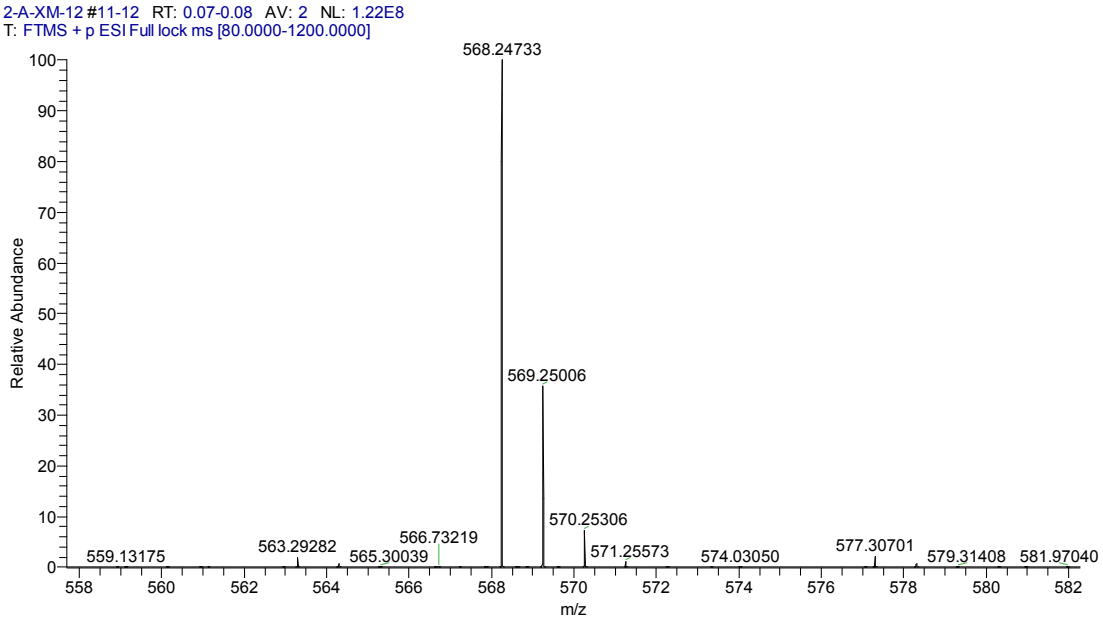
3ba


2-A-XM-10 #12 RT: 0.08 AV: 1 NL: 1.12E8
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
564.27252
565.27515
566.27808
562.76300
559.31793
561.32202
567.28125
569.12378
573.33203
575.33936
Relative Abundance
m/z

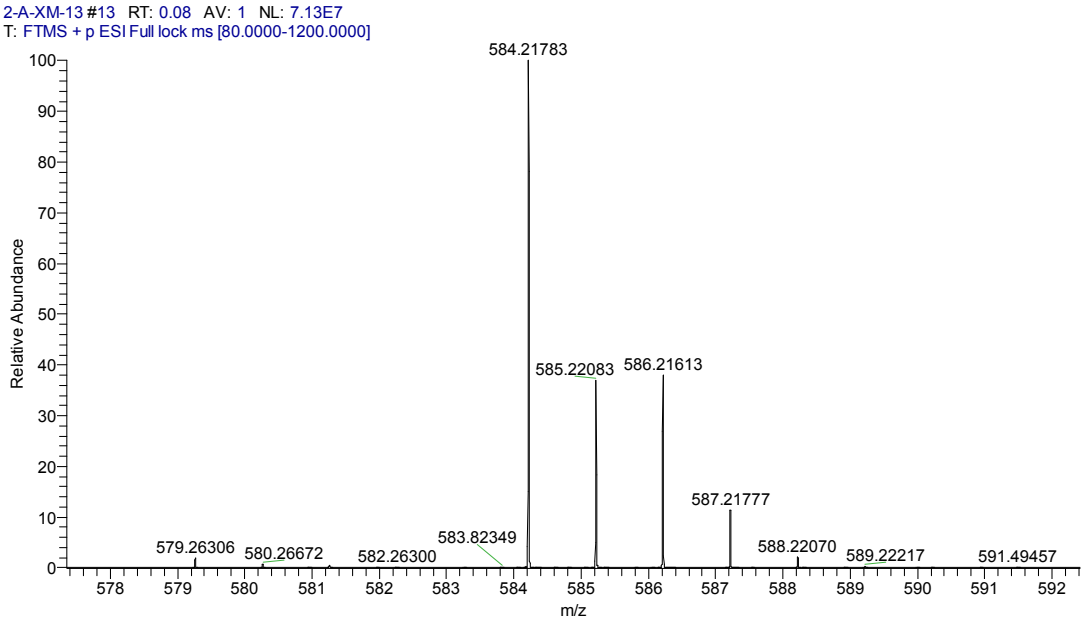
3ca


2-A-XM-11 #11 RT: 0.07 AV: 1 NL: 3.63E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
580.26727
581.27020
582.27319
575.31244
577.32019
579.86438
583.27594
586.05084
589.32703
Relative Abundance
m/z

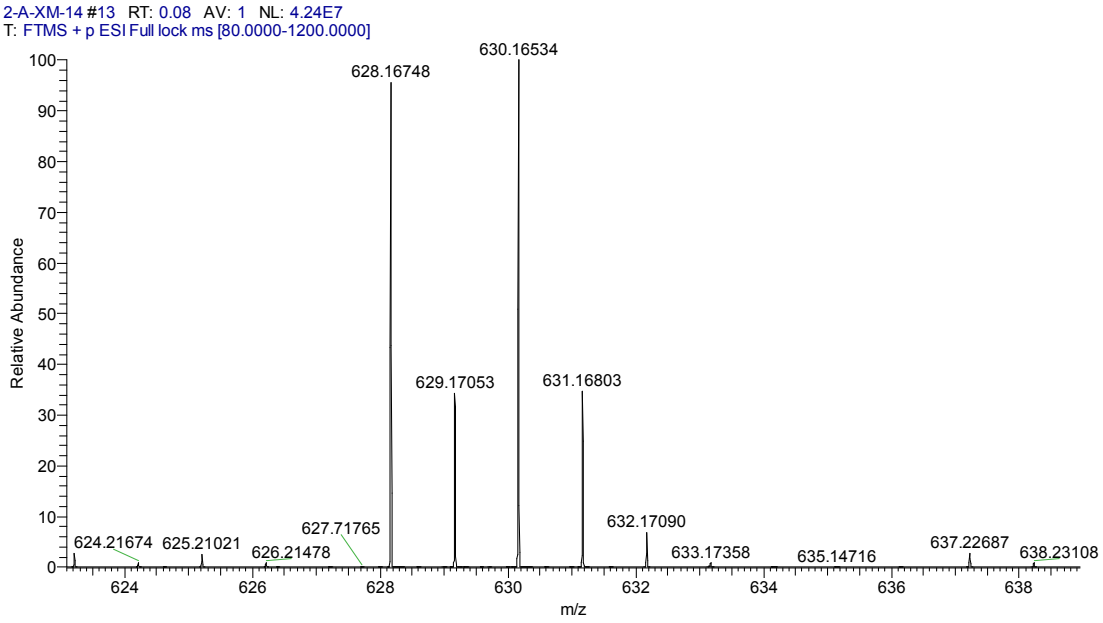
3da


2-A-XM-12 #11-12 RT: 0.07-0.08 AV: 2 NL: 1.22E8
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
568.24733
569.25006
570.25306
559.13175
563.29282
565.30039
566.73219
571.25573
574.03050
577.30701
579.31408
581.97040
Relative Abundance
m/z

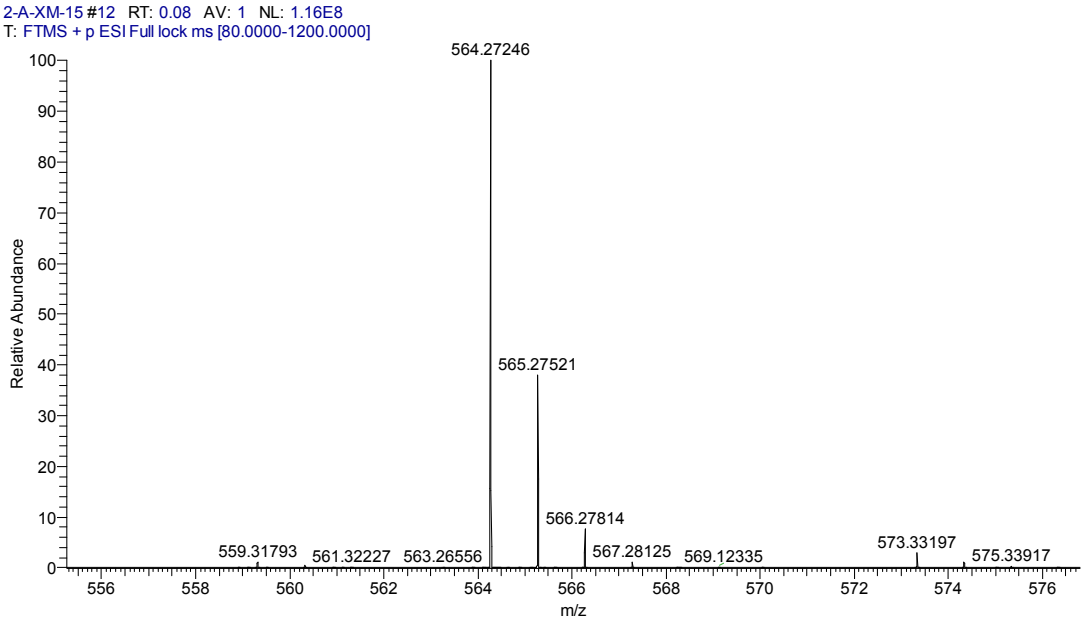
3ea


2-A-XM-13 #13 RT: 0.08 AV: 1 NL: 7.13E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
584.21783
585.22083
586.21613
587.21777
579.26306
580.26672
582.26300
583.82349
588.22070
589.22217
591.49457
Relative Abundance
m/z

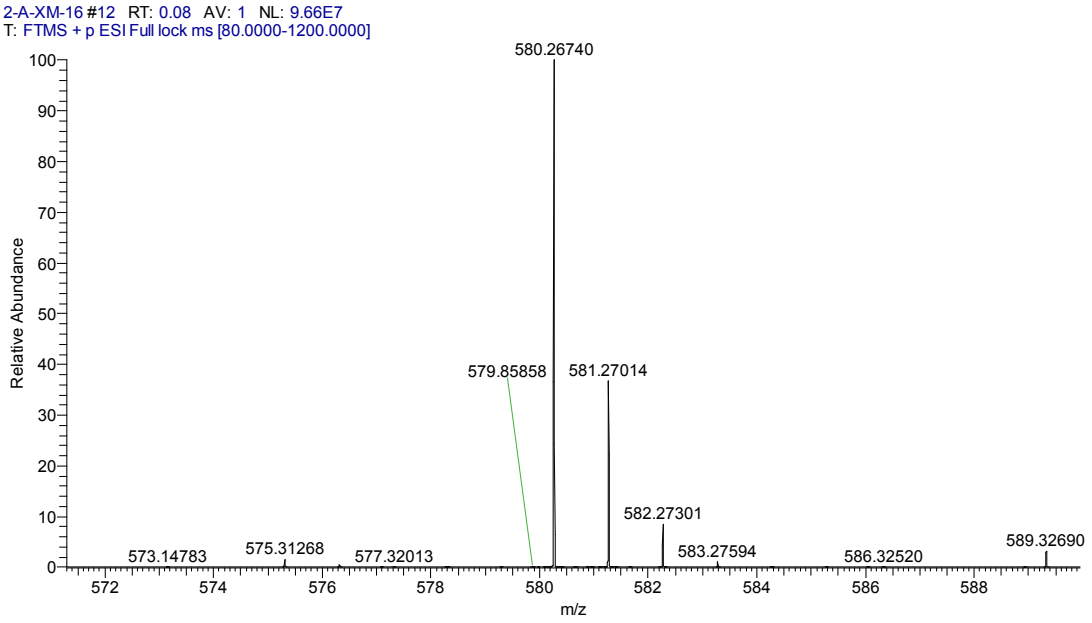
3fa


2-A-XM-14 #13 RT: 0.08 AV: 1 NL: 4.24E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
Relative Abundance
m/z
630.16534
628.16748
629.17053
631.16803
632.17090
624.21674
625.21021
626.21478
627.71765
633.17358
635.14716
637.22687
638.23108

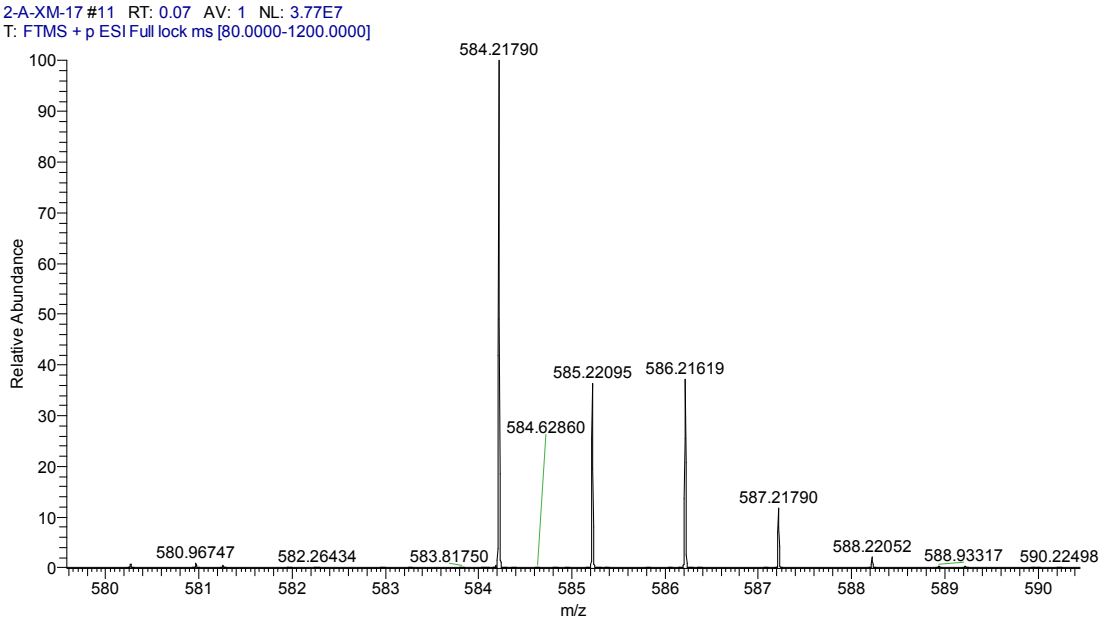
3ga


2-A-XM-15 #12 RT: 0.08 AV: 1 NL: 1.16E8
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
Relative Abundance
m/z
564.27246
565.27521
566.27814
559.31793
561.32227
563.26556
567.28125
569.12335
573.33197
575.33917

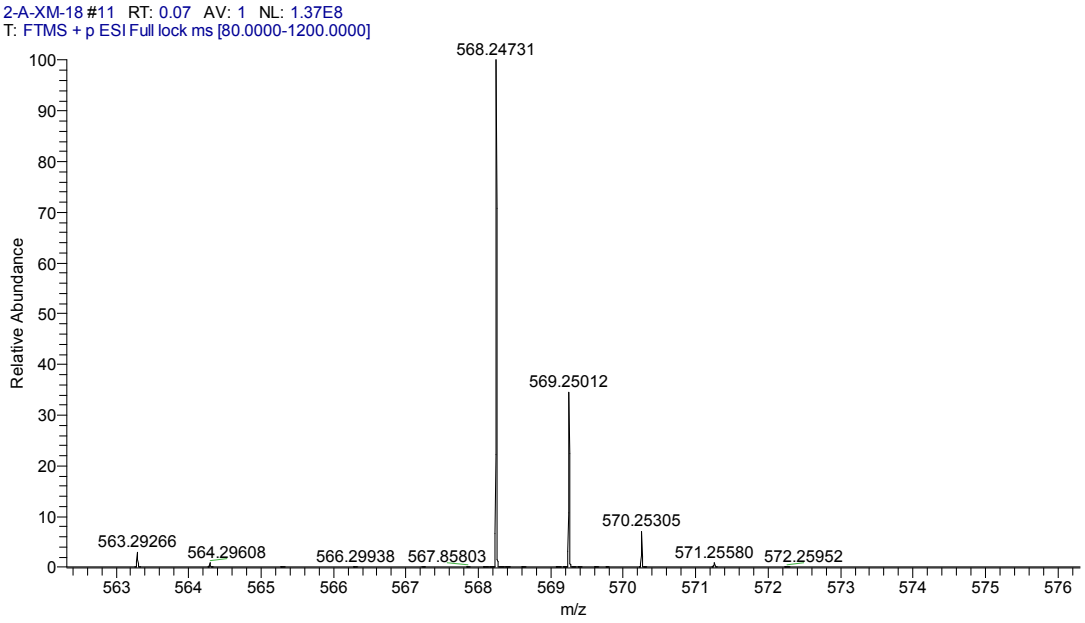
3ha


2-A-XM-16 #12 RT: 0.08 AV: 1 NL: 9.66E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
580.26740
579.85858
581.27014
582.27301
573.14783
575.31268
577.32013
583.27594
586.32520
589.32690
Relative Abundance
m/z

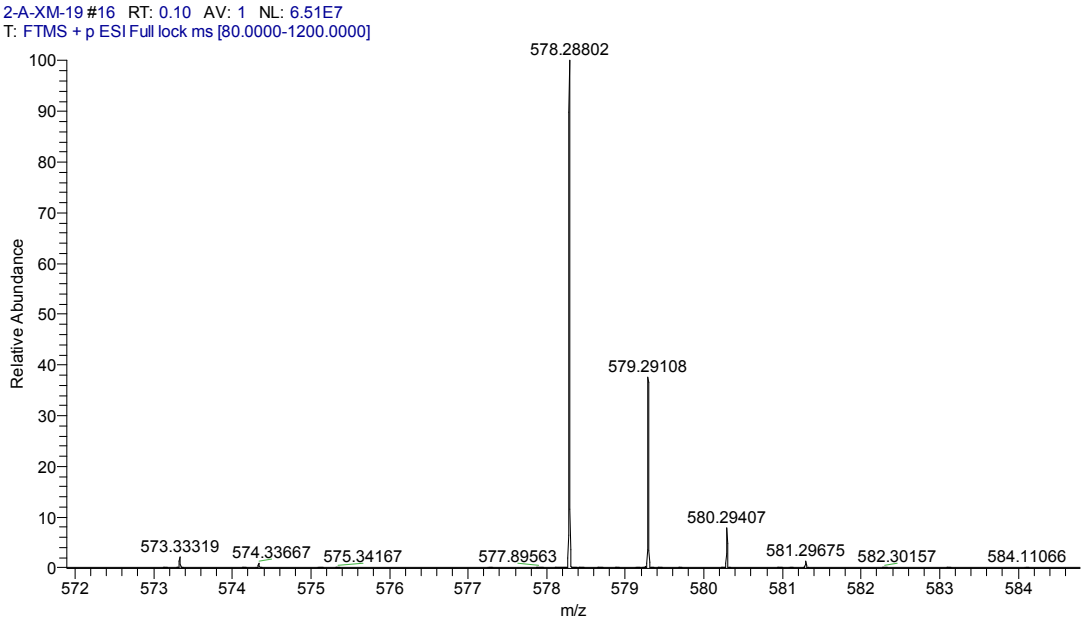
3ia


2-A-XM-17 #11 RT: 0.07 AV: 1 NL: 3.77E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
584.21790
585.22095
586.21619
584.62860
587.21790
580.96747
582.26434
583.81750
588.22052
588.93317
590.22498
Relative Abundance
m/z

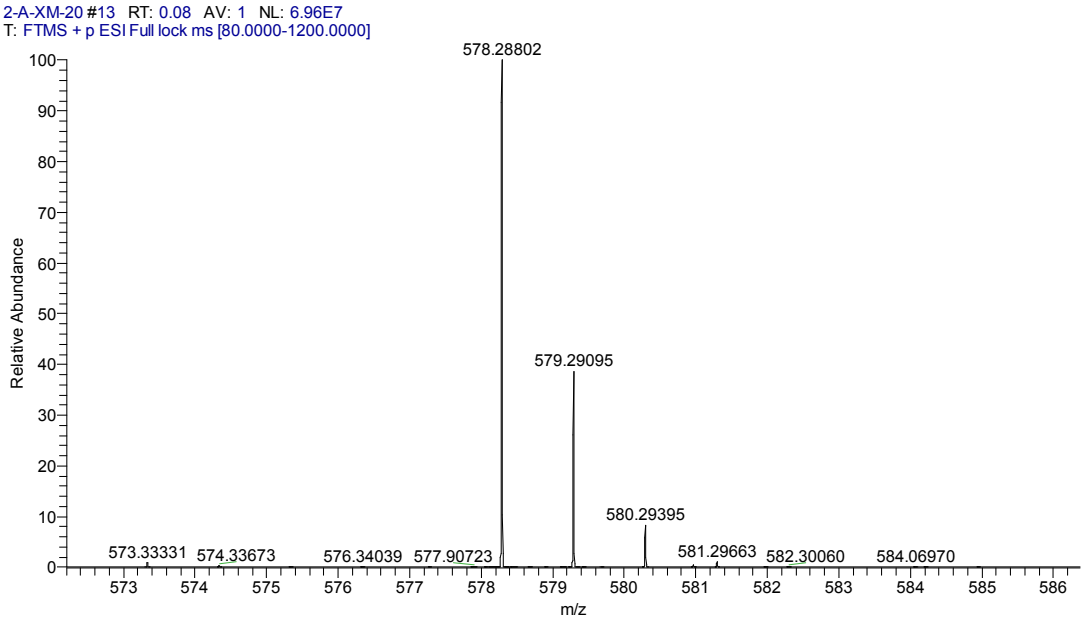
3ja


2-A-XM-18 #11 RT: 0.07 AV: 1 NL: 1.37E8
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
Relative Abundance
m/z
568.24731
569.25012
570.25305
563.29266
564.29608
566.29938
567.85803
571.25580
572.25952

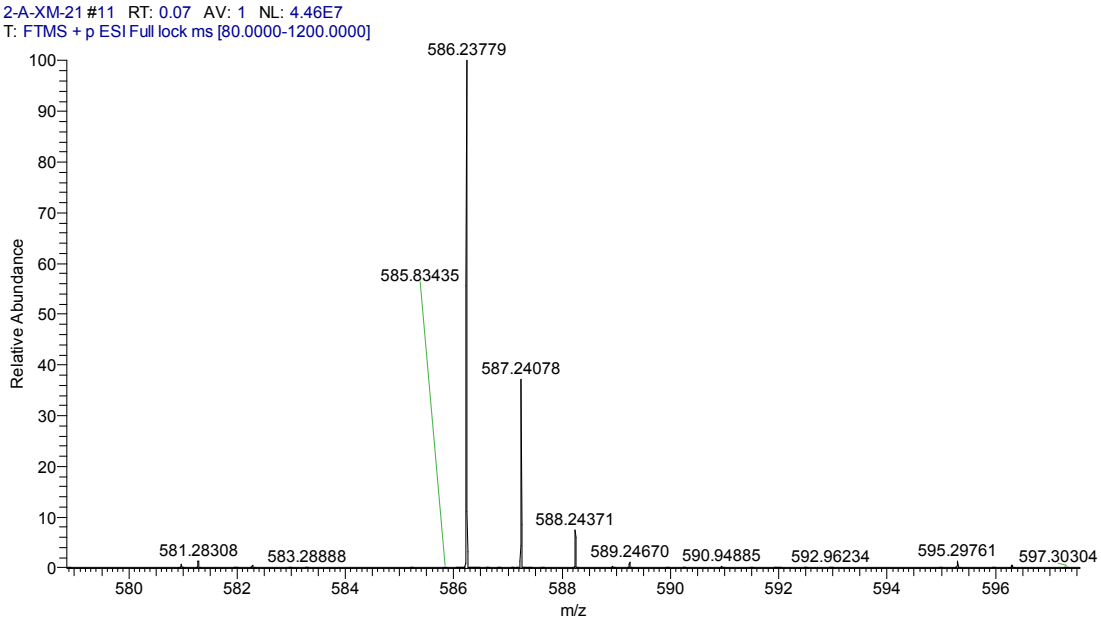
3ka


2-A-XM-19 #16 RT: 0.10 AV: 1 NL: 6.51E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
Relative Abundance
m/z
578.28802
579.29108
580.29407
573.33319
574.33667
575.34167
577.89563
581.29675
582.30157
584.11066

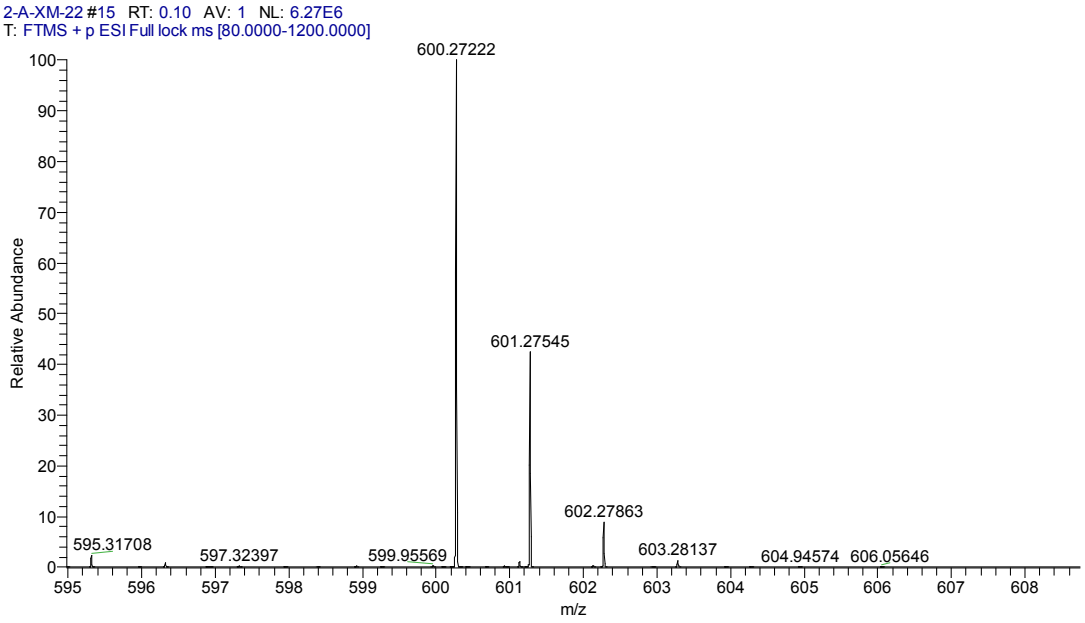
3la


2-A-XM-20 #13 RT: 0.08 AV: 1 NL: 6.96E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
578.28802
579.29095
580.29395
573.33331
574.33673
576.34039
577.90723
581.29663
582.30060
584.06970
Relative Abundance
m/z

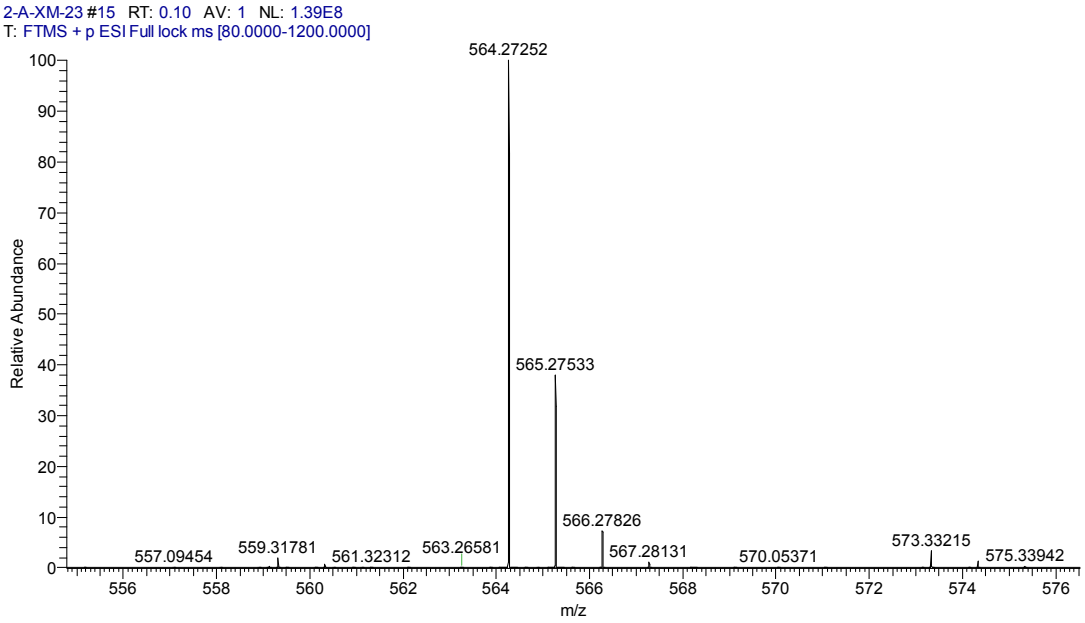
3ma


2-A-XM-21 #11 RT: 0.07 AV: 1 NL: 4.46E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
586.23779
585.83435
587.24078
588.24371
581.28308
583.28888
589.24670
590.94885
592.96234
595.29761
597.30304
Relative Abundance
m/z

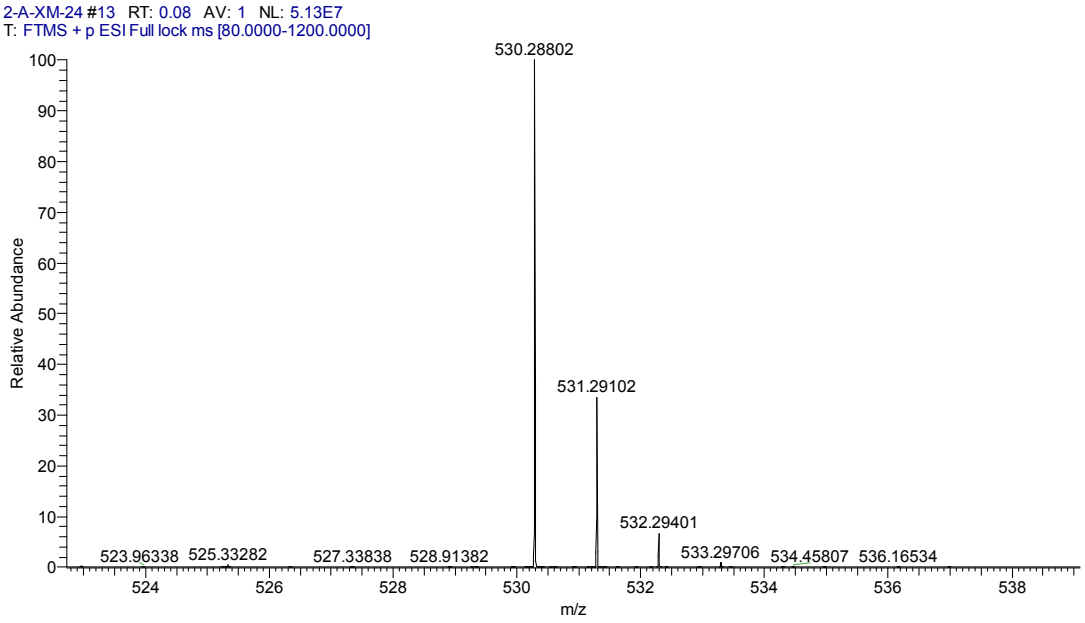
3na


2-A-XM-22 #15 RT: 0.10 AV: 1 NL: 6.27E6
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
600.27222
601.27545
602.27863
595.31708
597.32397
599.95569
603.28137
604.94574
606.05646
Relative Abundance
m/z

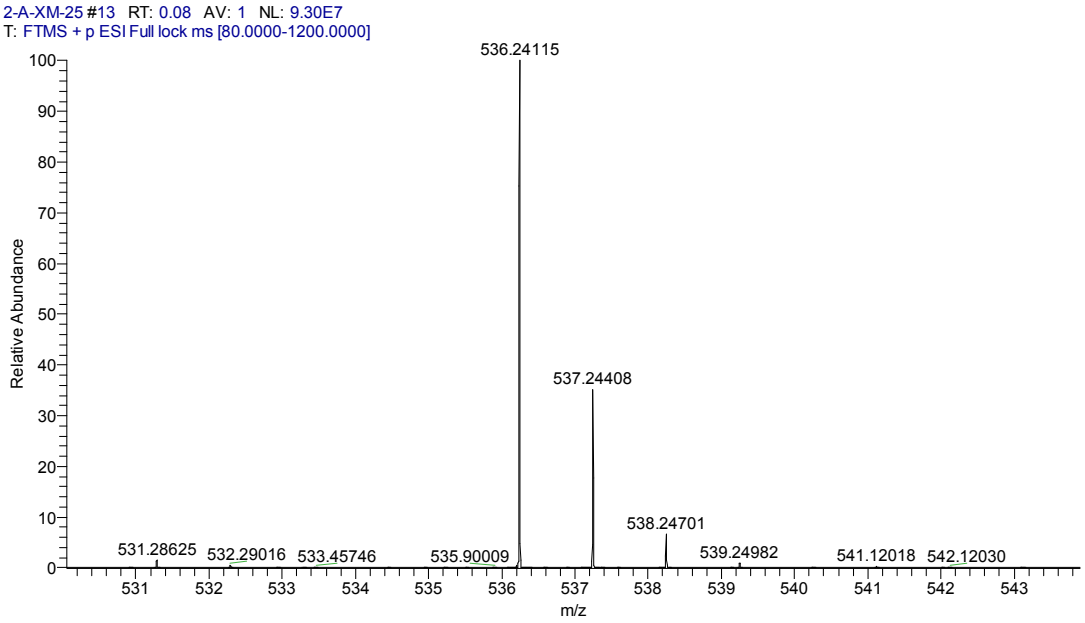
3oa


2-A-XM-23 #15 RT: 0.10 AV: 1 NL: 1.39E8
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
564.27252
565.27533
566.27826
557.09454
559.31781
561.32312
563.26581
567.28131
570.05371
573.33215
575.33942
Relative Abundance
m/z

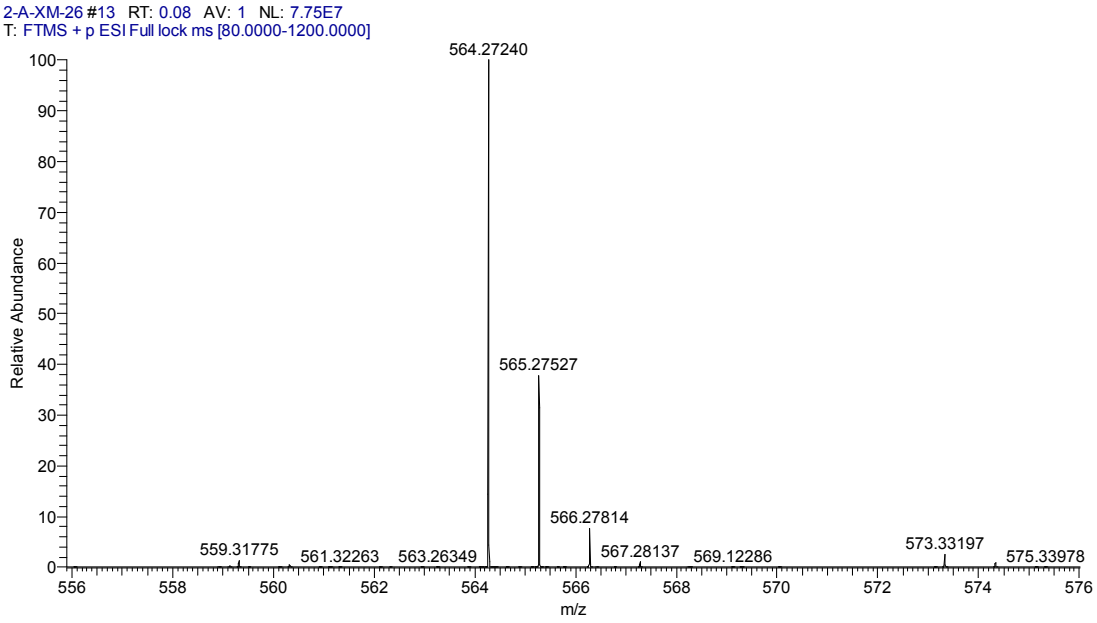
3pa


2-A-XM-24 #13 RT: 0.08 AV: 1 NL: 5.13E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
530.28802
531.29102
532.29401
533.29706
534.45807
536.16534
523.96338
525.33282
527.33838
528.91382
Relative Abundance
m/z

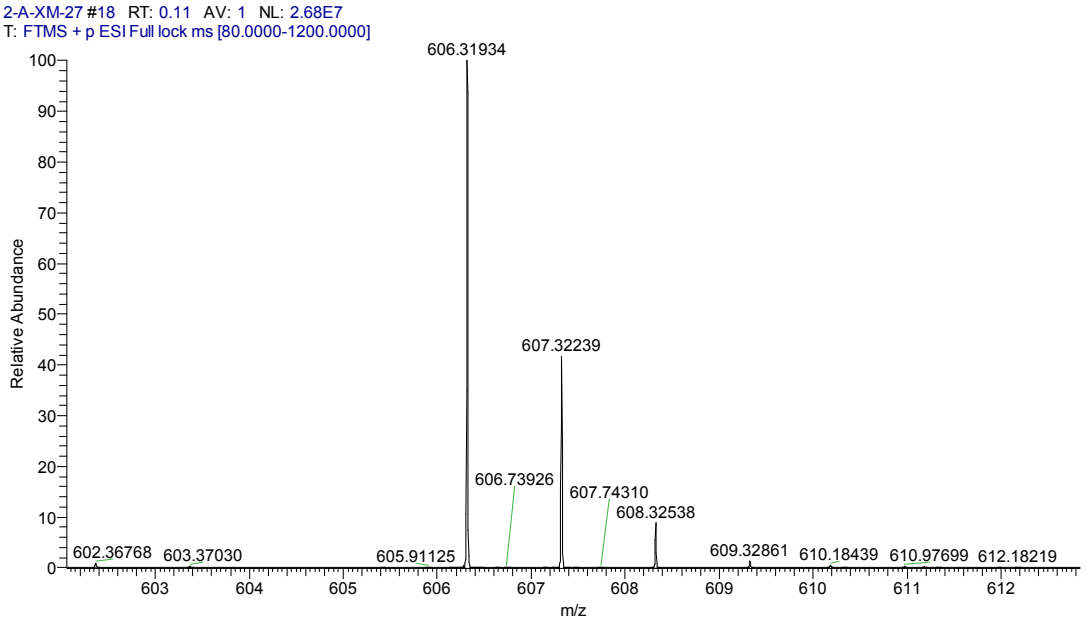
3qa


2-A-XM-25 #13 RT: 0.08 AV: 1 NL: 9.30E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
536.24115
537.24408
538.24701
539.24982
541.12018
542.12030
531.28625
532.29016
533.45746
535.90009
Relative Abundance
m/z

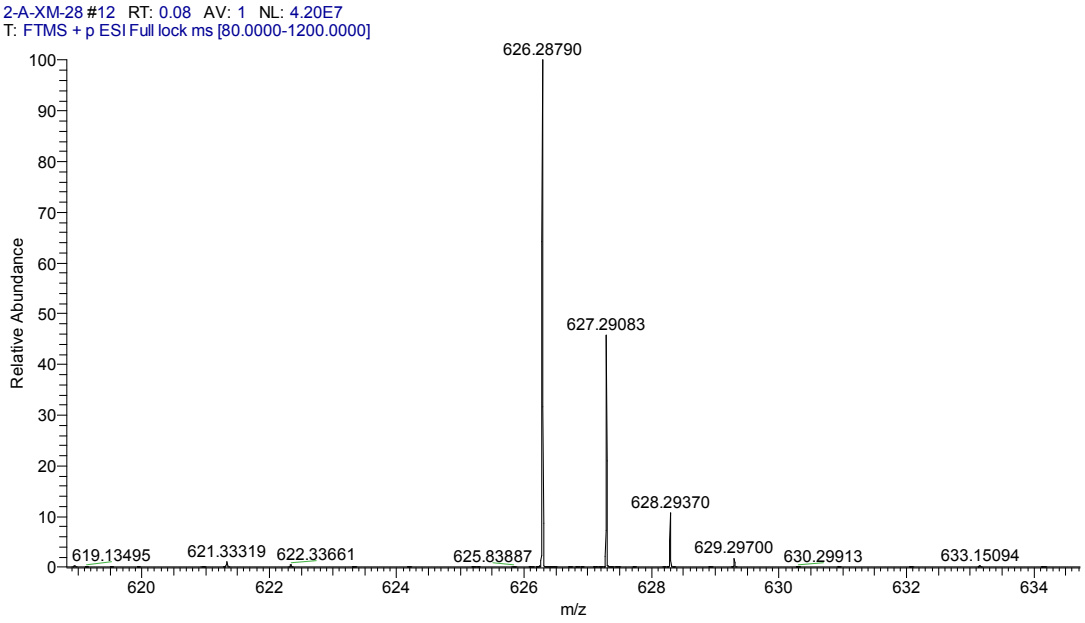
3ra


2-A-XM-26 #13 RT: 0.08 AV: 1 NL: 7.75E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
564.27240
565.27527
566.27814
559.31775
561.32263
563.26349
567.28137
569.12286
573.33197
575.33978
Relative Abundance
m/z

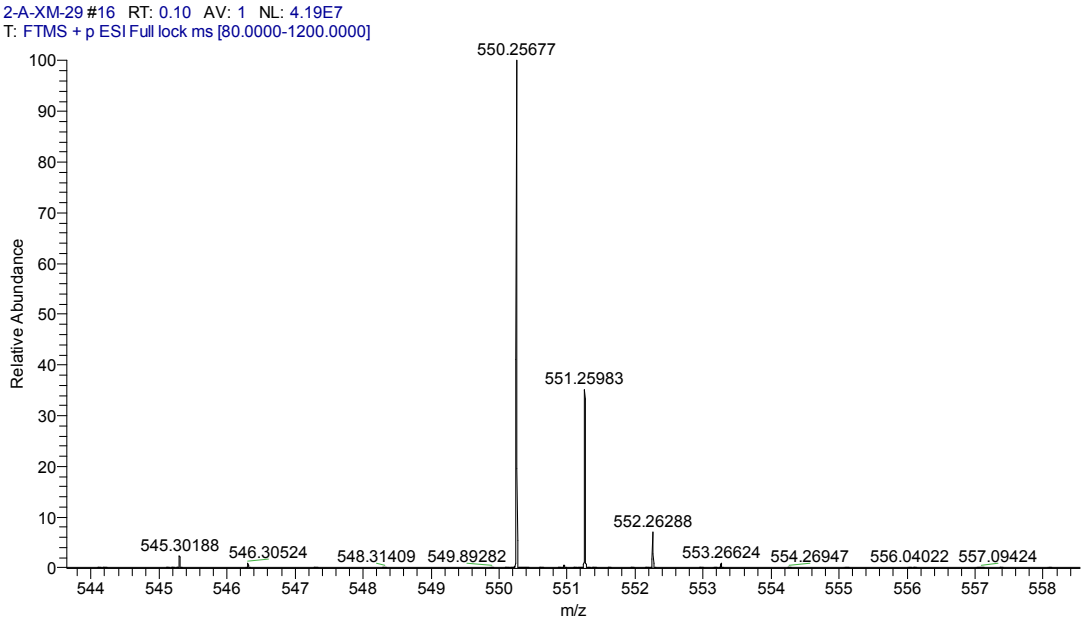
3sa


2-A-XM-27 #18 RT: 0.11 AV: 1 NL: 2.68E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
606.31934
607.32239
606.73926
607.74310
608.32538
602.36768
603.37030
605.91125
609.32861
610.18439
610.97699
612.18219
Relative Abundance
m/z

3ta


2-A-XM-28 #12 RT: 0.08 AV: 1 NL: 4.20E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
626.28790
627.29083
628.29370
629.29700
630.29913
633.15094
619.13495
621.33319
622.33661
625.83887
Relative Abundance
m/z

6


2-A-XM-29 #16 RT: 0.10 AV: 1 NL: 4.19E7
T: FTMS + p ESI Full lock ms [80.0000-1200.0000]
550.25677
551.25983
552.26288
553.26624
554.26947
556.04022
557.09424
545.30188
546.30524
548.31409
549.89282
Relative Abundance
m/z