

Supporting Information

Assembly of Protein-mimetic Helical Chiral Metal-Organic Framework for Enantioseparation

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1. Materials and general procedures.

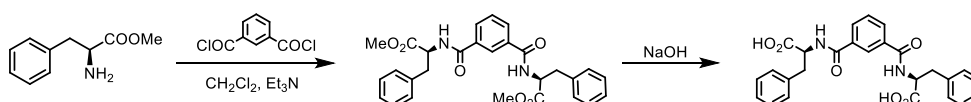
All of the chemicals are commercially available and used without any further purification. Thermogravimetric analysis of (*S*)-**1** was carried out in a nitrogen atmosphere with a heating rate of 10 °C/min on a TGA-50 thermogravimetric analyzer. Powder X-ray diffraction (PXRD) data were collected on a DMAX2500 diffractometer using Cu K α radiation. Simulated PXRD patterns were generated from the single-crystal structural data of (*S*)-**1** using the XPow module of the SHELXTL software package. The dye adsorption experiment was monitored using an Agilent Technologies Cary UV/Vis Spectrometer. The infrared (IR) spectrum (KBr pellet) was recorded (500-4000 cm⁻¹ region) on a Nicolet Magna 750 Fourier transform infrared (FTIR) spectrometer. Analytical high-performance liquid chromatography (HPLC) was performed on an Agilent Technologies 1260 Infinity II system equipped with a UV-vis detector. Analytical ChiralCel OD-H/OD/OJ-H/AS-H/IC-H/AD-H/IA-H columns (4.6 mm \times 25 cm) from Daicel were used.

X-ray Crystallography: Single-crystal X-ray diffraction (SC-XRD) data for chiral MOFs, (*S*)-**1** and (*R*)-**1**, were collected at 100 K on BL17B beamline of the National Facility for Protein Science (NFPS, Shanghai) using synchrotron radiation, with wavelengths of 0.71253 Å and 0.68883 Å, respectively. All of the diffraction datasets were processed through indexing, integration, and scaling with the APEX4 program. The structures of (*S*)-**1** and (*R*)-**1** were solved by direct method and refined *via* full-matrix least-squares technique on F^2 using the SHELXS-2014 within the Olex2 interface. All non-hydrogen atoms belonging to the flexible ligands were refined with anisotropic displacement parameters, while all ligand-attached hydrogen atoms were placed in calculated positions and refined using a riding model. Contributions to scattering due to these highly disordered guest molecules in (*S*)-**1** and (*R*)-**1** were removed using the SQUEEZE subroutine of the PLATON software package. The structures were then refined again using the resulting new HKL file. (*S*)-**1** and (*R*)-**1** can be best formulated as [ZnL(TMDP)], on the basis of single-crystal diffraction, IR spectra, UV-vis spectra, and thermogravimetric analyses (TGA). Crystal data and details of the data collection are given in Table S1, while the selected bond distances and angles are presented in Table S2. CCDC numbers are 2535054 for (*S*)-**1** and 2535261 for (*R*)-**1**, respectively, which contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.

Enantioseparation procedure: Prior to the enantioselective sorption experiment, the pristine crystals of (*S*)-**1** and (*R*)-**1** were treated three times with anhydrous methanol and acetone, respectively, to exchange the guest solvent molecules (e.g., DMF) within their chiral pores. Subsequently, the solvent-exchanged crystals (ca. 100 mg) were immersed in a 2 mL solution of indicated racemic analyte (10 mM) for 12 h at room temperature. Afterward, the solid sample was filtered and washed several times with fresh solvent to remove the racemic analytes adsorbed on the crystal surfaces. Finally, the solid samples were immersed in acetone to extract the chiral epoxides encapsulated within their channels. The enantiomeric purity of the extracted chiral guest was determined by HPLC.

Dye adsorption: About 50 mg solvent-exchanged crystals of (*S*)-**1** were immersed in 2.5 mL ethanol solution of 50 μ M dye molecule, 4-chloroisatin. Then the 4-chloroisatin content in solution was monitored by UV-Vis at different time periods.

2. Synthesis of chiral ligand (*S*)-H₂L and chiral MOFs (*S*)-1 and (*R*)-1.



(*S*)-H₂L was synthesized from optically pure *L*-phenylalanine methyl ester according to the above reaction routine. *L*-phenylalanine methyl ester (3 g, 16.74 mmol) was dissolved in dichloromethane (60 mL), followed by the dropwise addition of isophthaloyl chloride (1.70 g, 8.37 mmol) and Et₃N (10.0 mL). The resulting reaction mixture was stirred at room temperature for an additional 6 h. After that, the mixture was washed with water (3 × 20 mL), 2 M HCl (3 × 20 mL), and a saturated NaHCO₃ solution (3 × 20 mL). Subsequently, the organic phase was dried over Na₂SO₄ and concentrated under reduced pressure, affording the methylated chiral ligand, *L*-Me₂ (ca. 3.87 g), with a yield of ca. 94.6%.

The hydrolysis of *L*-Me₂ (3.87 g, 7.92 mmol) was carried out in a mixed alkaline solution of NaOH (0.32 g, 79.22 mmol) in MeOH/THF/H₂O (20 mL/20 mL/5 mL) at room temperature for 6 h. The resulting mixture was acidified with 2 M HCl and filtered. The product was extracted from the aqueous phase with ethyl acetate. The organic phase was dried over Na₂SO₄ and concentrated under reduced pressure to give the chiral ligand (*S*)-H₂L (3.33 g) in a yield of ca. 91.3%. (*R*)-H₂L was synthesized using the identical procedure, but employing *D*-phenylalanine as the chiral source.

A mixture of Zn(NO₃)₂·6H₂O (14.5 mg, 0.05 mmol), H₂L (18.4 mg, 0.05 mmol) and TMDP co-ligand (8 mg, 0.05 mmol) was placed in a glass vial containing DMF (2.5 mL), MeCN (2.5 mL). The vial was sealed tightly and heated at 60 °C for three days. Colorless block crystals of [ZnL(TMDP)] ((*S*)-1) were generated. The crystals of (*R*)-1 can be obtained under the same synthetic condition as that of (*S*)-1 except the (*S*)-H₂L was replaced by (*R*)-H₂L.

3. Table S1. Crystal data and structure refinement for (*S*)-1 and (*R*)-1.

Empirical formula	C ₃₉ H ₃₆ N ₄ O ₆ Zn	C ₃₉ H ₃₆ N ₄ O ₆ Zn
Formula weight	722.09	722.09
Temperature (K)	100	100
Identification code	(<i>S</i>)-1	(<i>R</i>)-1
Wavelength (Å)	0.71253	0.68883
Crystal system, space group	Trigonal, <i>P</i> 3 ₂ 21	Trigonal, <i>P</i> 3 ₁ 21
Unit cell dimensions	a = 18.5336(1) Å b = 18.5336(1) Å c = 23.6582(1) Å α = 90°, β = 90°, γ = 120°.	a = 18.5346(1) Å b = 18.5346(1) Å c = 23.6007(1) Å α = 90°, β = 90°, γ = 120°.
Volume	7037.7(1) Å ³	7021.4(1) Å ³
Z, Calculated density	6, 1.022 mg/m ³	6, 1.025 mg/m ³
Absorption coefficient	0.563 mm ⁻¹	0.522 mm ⁻¹
F(000)	2256	2256
θ range for data collection (°)	2.144 to 24.779	1.487 to 29.837
Limiting indices	-21 ≤ h ≤ 21, -21 ≤ k ≤ 21, -27 ≤ l ≤ 27	-24 ≤ h ≤ 24, -26 ≤ k ≤ 26, -30 ≤ l ≤ 29
Reflections	86189/7945, Rint = 0.0785	102754/13097, Rint = 0.0465

collected/independent reflections		
Completeness to theta	99.00%	99.00%
Refinement method	Full-matrix least-squares on F ²	Full-matrix least-squares on F ²
Data / restraints / parameters	7945 / 0 / 451	13097 / 0 / 451
Goodness-of-fit on F ²	1.072	1.027
Final R indices [I>2sigma(I)]	R1 = 0.0531, wR2 = 0.1218	R1 = 0.0596, wR2 = 0.1591
R indices (all data)	R1 = 0.0668, wR2 = 0.1259	R1 = 0.0569, wR2 = 0.1621
Absolute structure parameter	0.070(5)	0.141(4)

4. Table S2. Selected bond lengths [Å] and angles [°] for (S)-1 and (R)-1.

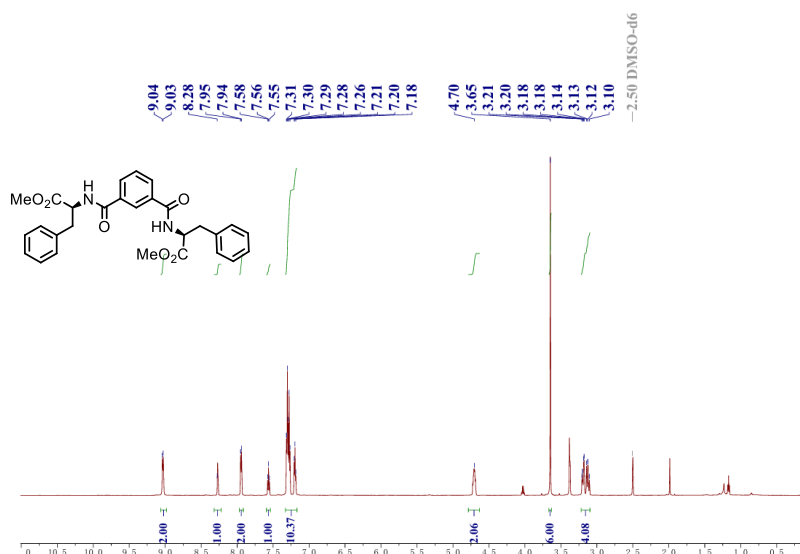
Bond Lengths	(S)-1	(R)-1
Zn(1)-O(1)#1	1.853(7)	1.948(3)
Zn(1)-O(6)	1.866(8)	1.942(2)
Zn(1)-N(3)#2	1.960(1)	2.002(3)
Zn(1)-N(4)	1.962(8)	2.062(3)
Bond Angles	(S)-1	(R)-1
O(1)#1-Zn(1)-O(6)	103.6(1)	104.09(11)
O(1)#1-Zn(1)-N(3)#2	110.7(5)	120.87(11)
O(1)#1-Zn(1)-N(4)	110.6(1)	105.07(12)
O(6)-Zn(1)-N(3)#2	120.7(6)	109.51(13)
O(6)-Zn(1)-N(4)	104.7(6)	113.03(12)
N(3)#2-Zn(1)-N(4)	105.2(7)	104.46(13)

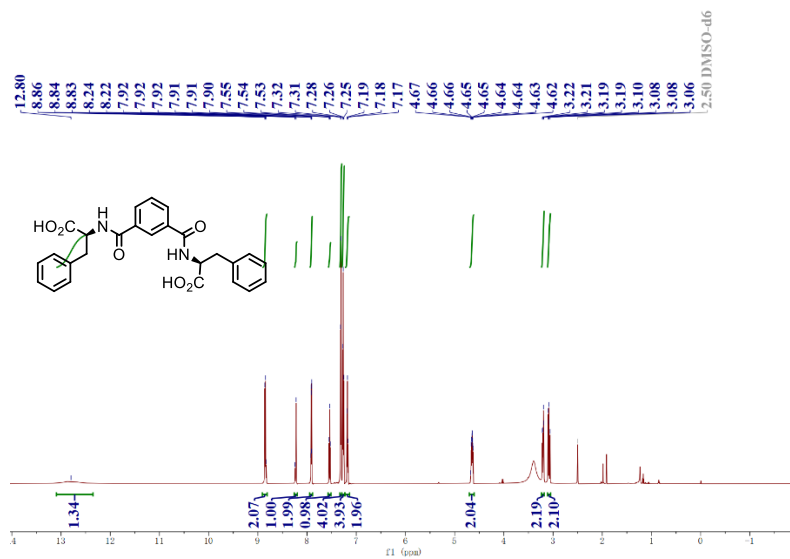
Symmetry transformations used to generate equivalent atoms:

(S)-1: #1 x, y, z, #2 -y, x-y, 2/3+z, #3 -x+y, -x, 1/3+z, #4 x-y, -y, 1/3-z, #5 -x, -x+y, 2/3-z, #6 y, x, -z

(R)-1: #1 x, y, z, #2 -y, x-y, z+1/3, #3 -x+y, -x, z+2/3, #4 y, x, -z, #5 x-y, -y, -z+2/3, #6 -x, -x+y, -z+1/3

5. Figures S1. ¹HNMR of L-Me₂ and H₂L





6. Figures S2-S4. Additional X-ray crystallographic structure of (S)-1.

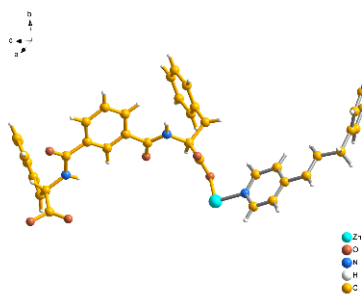


Figure S2. Asymmetric unit of (S)-1.

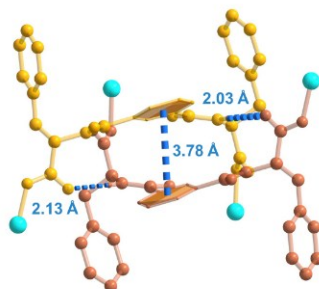


Figure S3. N-H...O hydrogen bonds and π ... π stackings between the adjacent 3_2 helical chains.

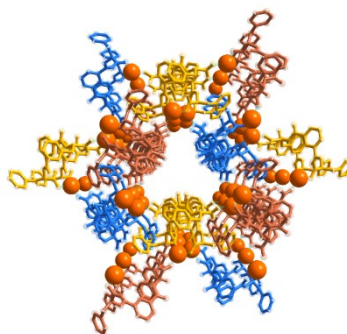
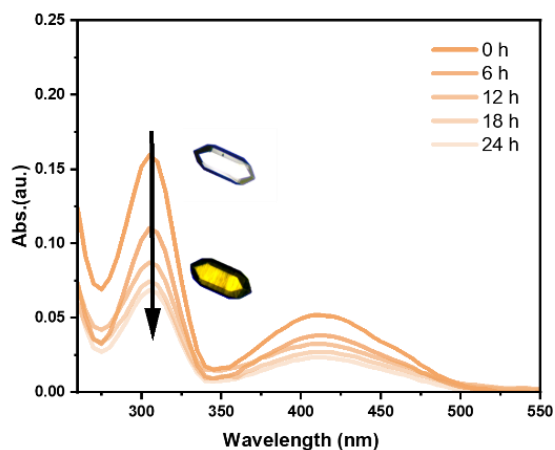
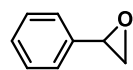


Figure S4. Helical nanochannels assembled from six 3_2 helices via non-covalent interactions.

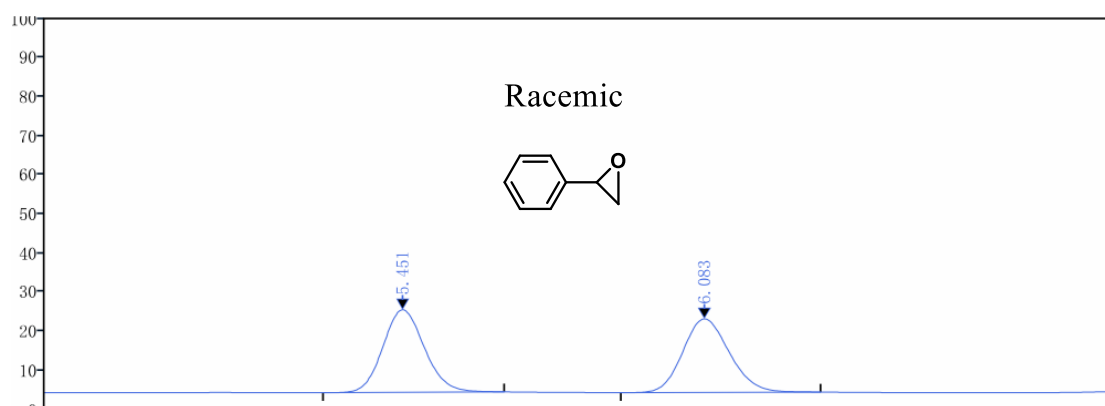
7. Figure S5. Adsorption of 4-chloroisatin by (*S*)-1 monitored by UV-Vis



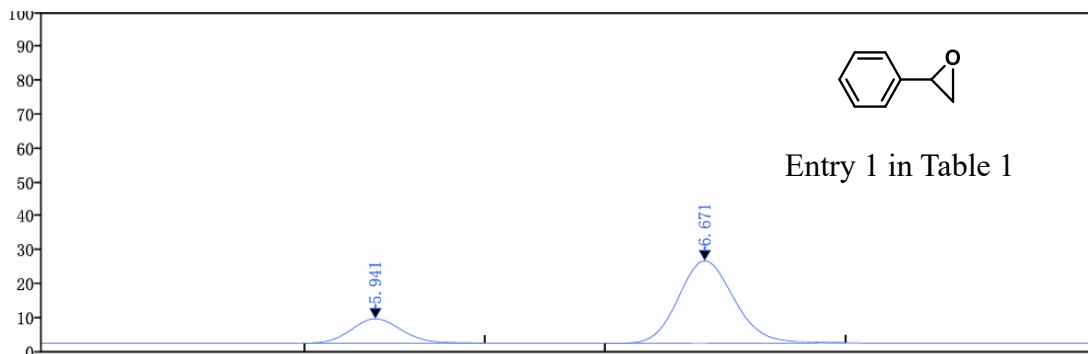
8. Figure S6. HPLC spectra of SO resolved by (*S*)-1 in different solvents (in Table 1).



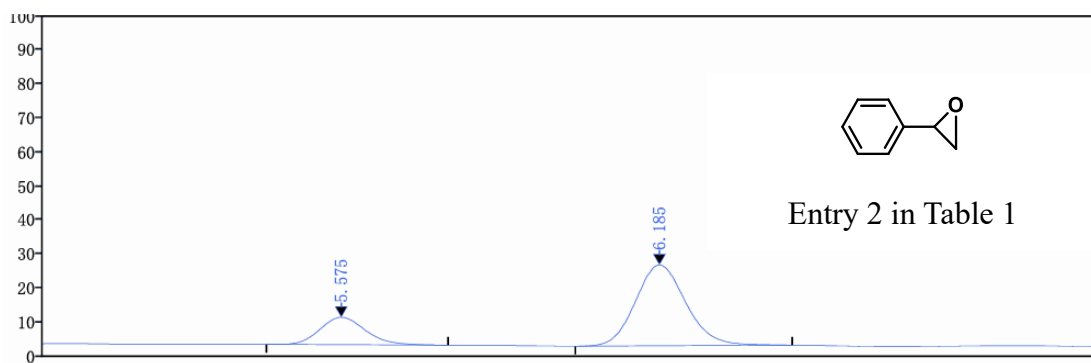
Chiral HPLC analysis: Daicel Chiralce IC: hexane/*i*-PrOH =88/12; flow rate =1.0 mL/min; 220 nm.



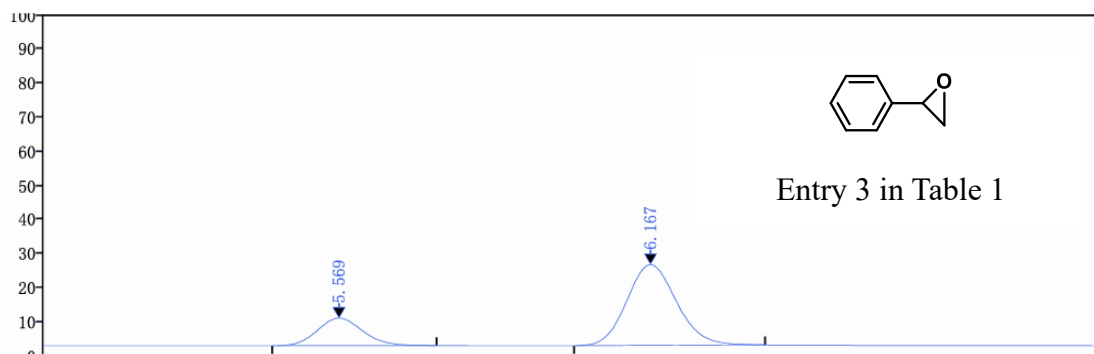
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.451	1597.55	50.02
2	MM	6.083	1596.26	49.98
The Total			3193.81	



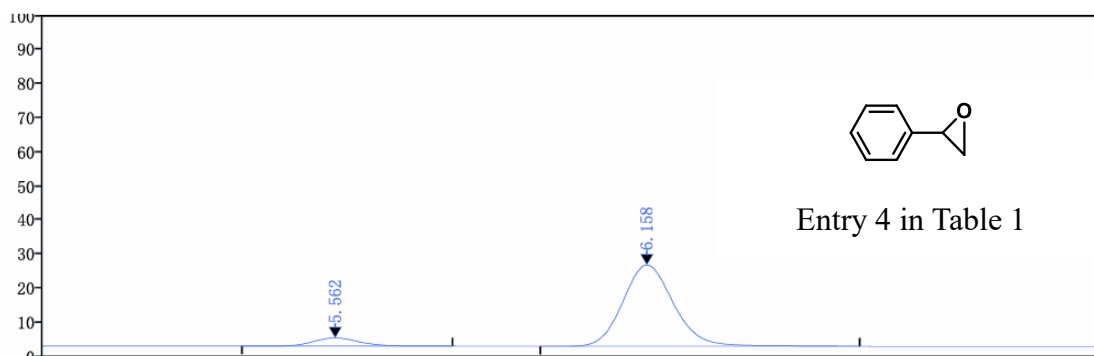
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.941	891.81	21.15
2	MM	6.671	3324.07	78.85
The Total			4215.88	



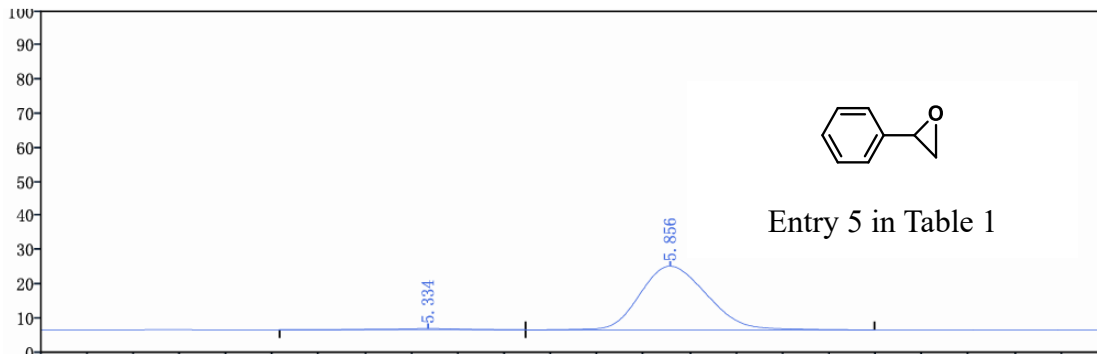
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.575	39.5	23.44
2	MM	6.185	128.98	76.56
The Total			168.48	



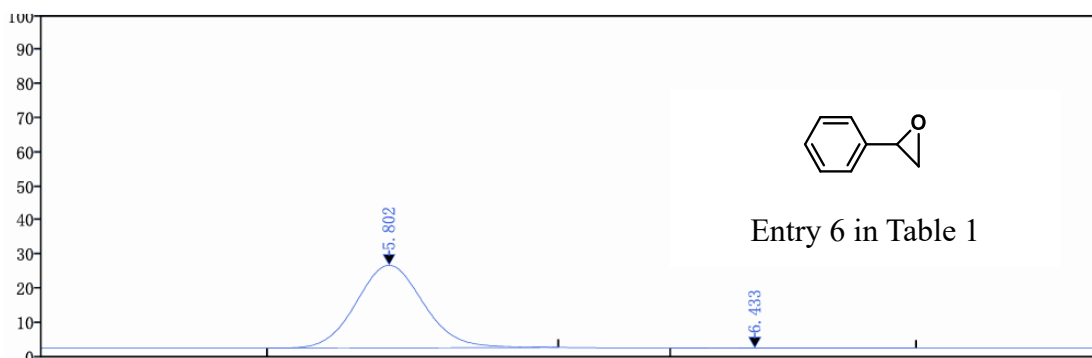
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.569	456.81	23.58
2	MM	6.167	1480.54	76.42
The Total			1937.35	



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.562	105.07	8.17
2	MM	6.158	1180.98	91.83
The Total			1286.05	

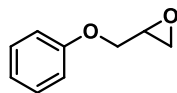
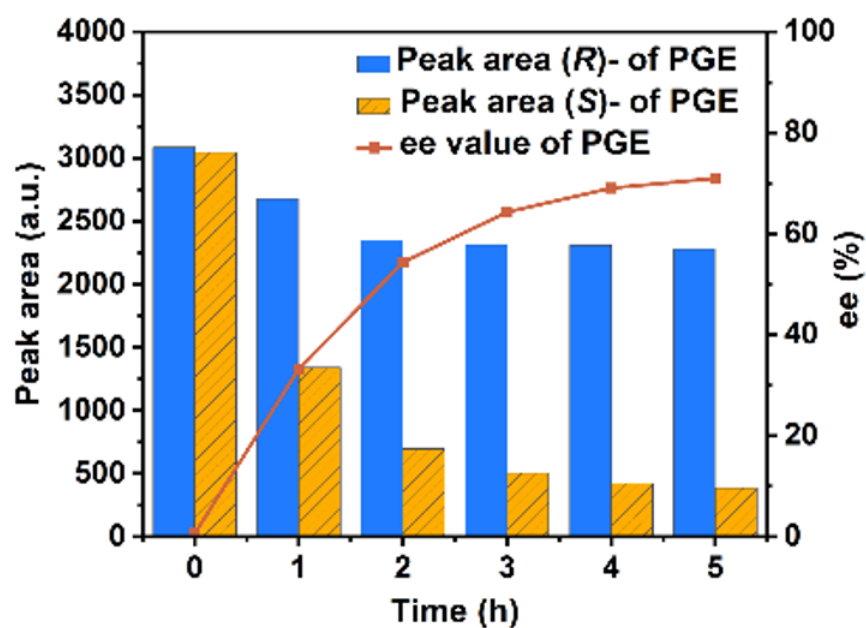


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.334	2.64	0.30
2	MM	5.856	873.60	99.70
The Total			876.24	

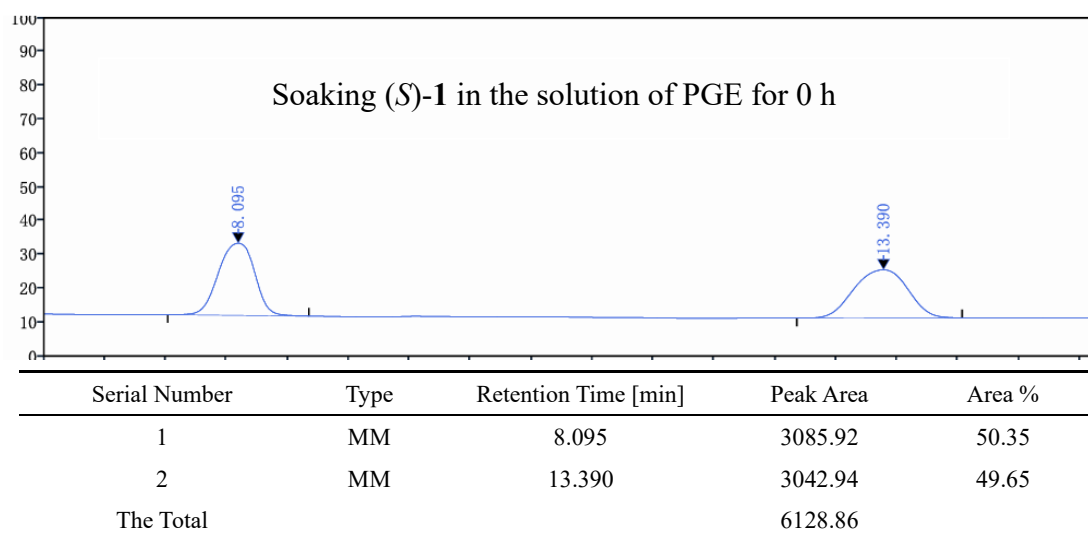


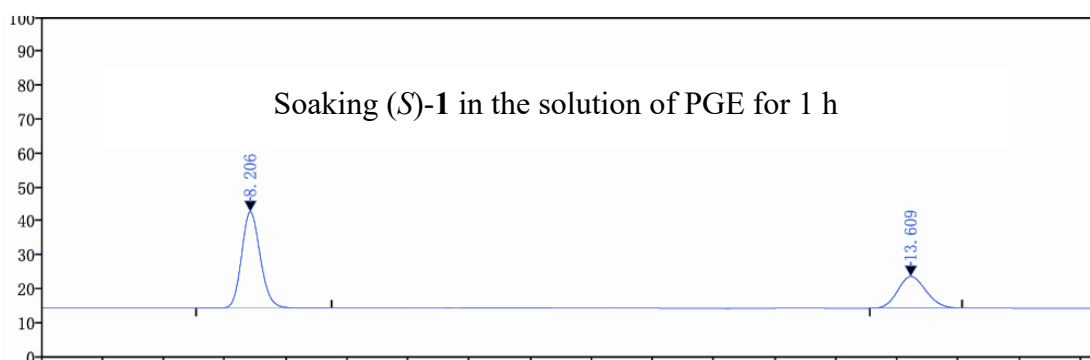
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.802	2366.50	99.80
2	MM	6.433	4.78	0.20
The Total			2371.28	

9. Figure S7. HPLC spectra of dynamic enantiosorption of (*S*)-1 towards PGE

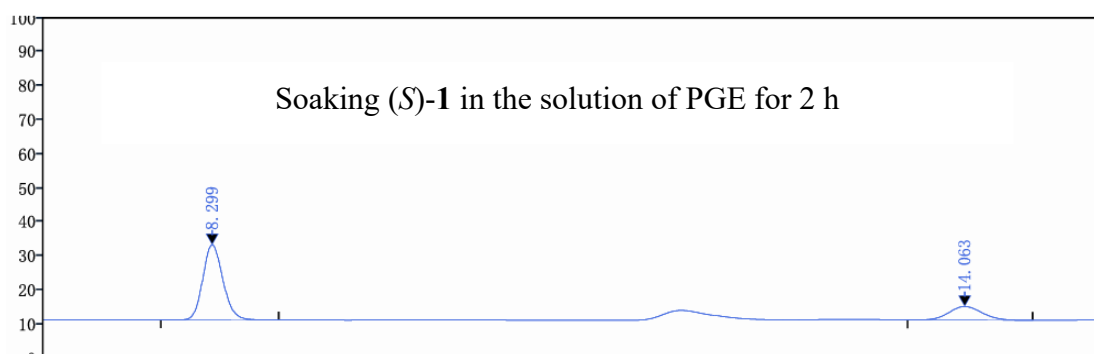


Chiral HPLC analysis: Daicel Chiralce OD-H: hexane/*i*-PrOH =90/10; flow rate =0.8 mL/min; 220 nm; $t_R = 8.095$ min, $t_R = 13.390$ min.

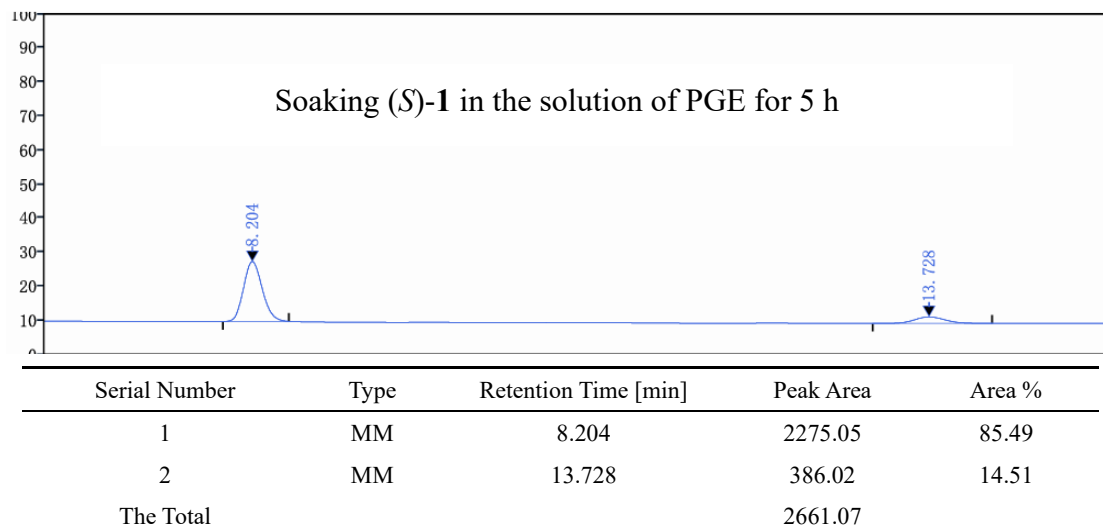
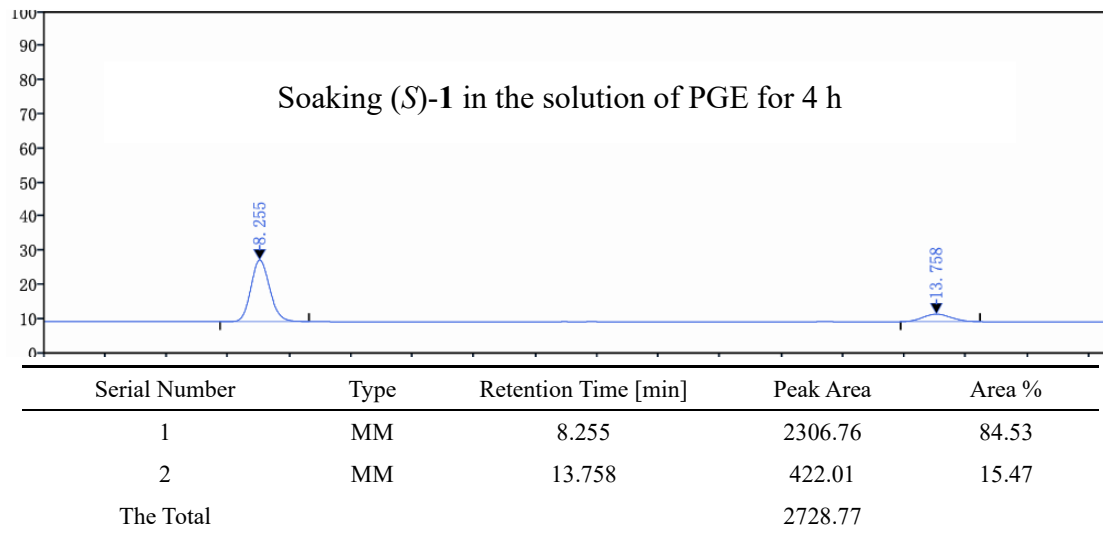
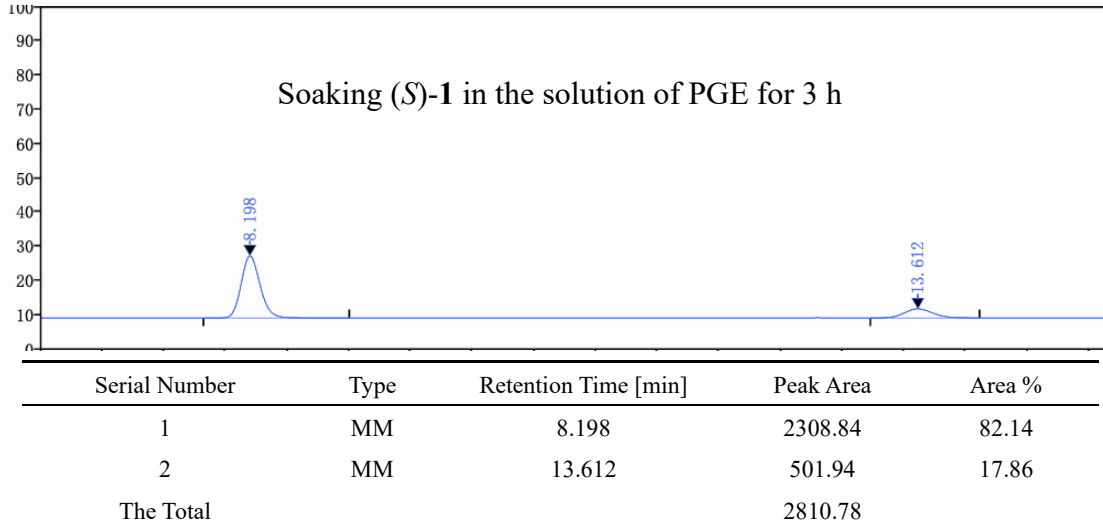




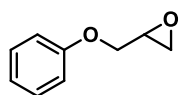
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.206	2674.37	66.61
2	MM	13.609	1340.68	33.39
The Total			4015.05	



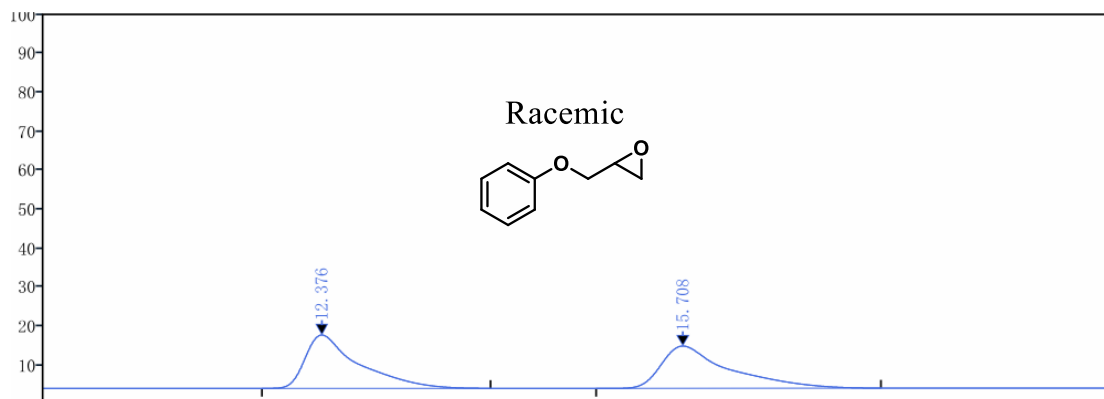
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.299	2345.15	77.12
2	MM	14.063	695.84	22.88
The Total			3040.99	



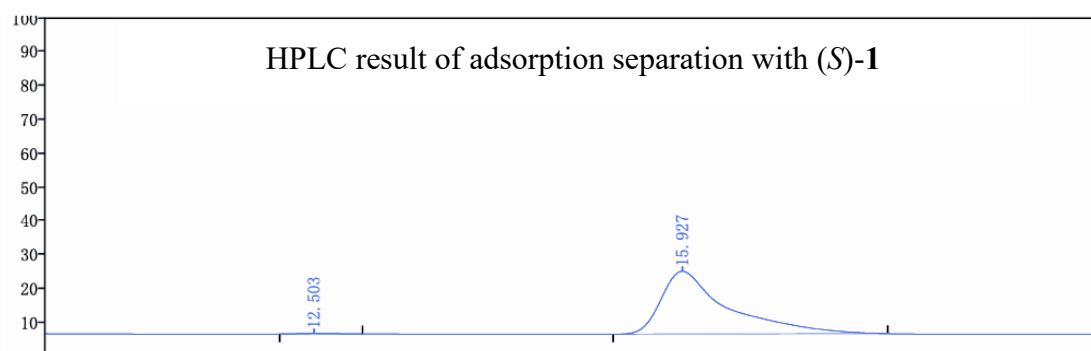
10. Figure S8. HPLC spectra of epoxides (in Figure 4).



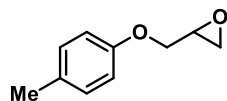
Chiral HPLC analysis: Daicel Chiralce AS-H: hexane/*i*-PrOH =98/2; flow rate =0.8 mL/min; 220 nm; $t_R = 12.376$ min, $t_R = 15.708$ min.



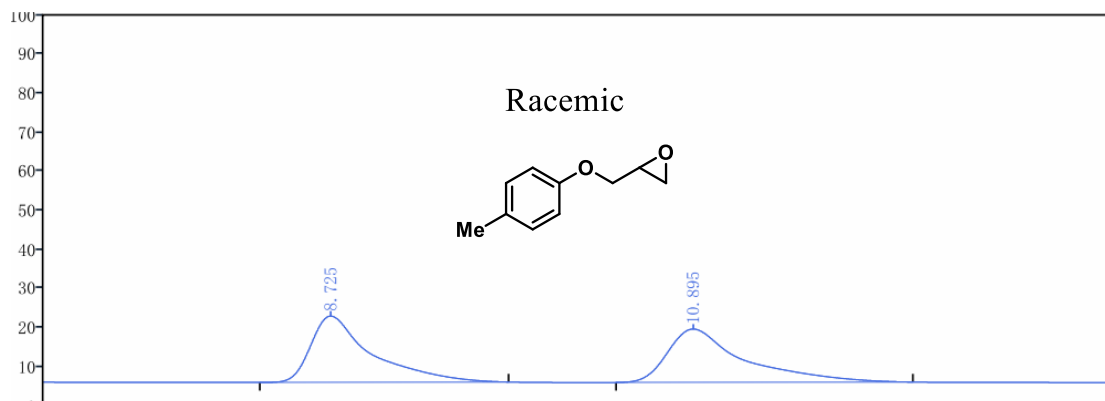
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	12.376	2777.10	49.93
2	MM	15.708	2785.04	50.07
The Total			5562.14	



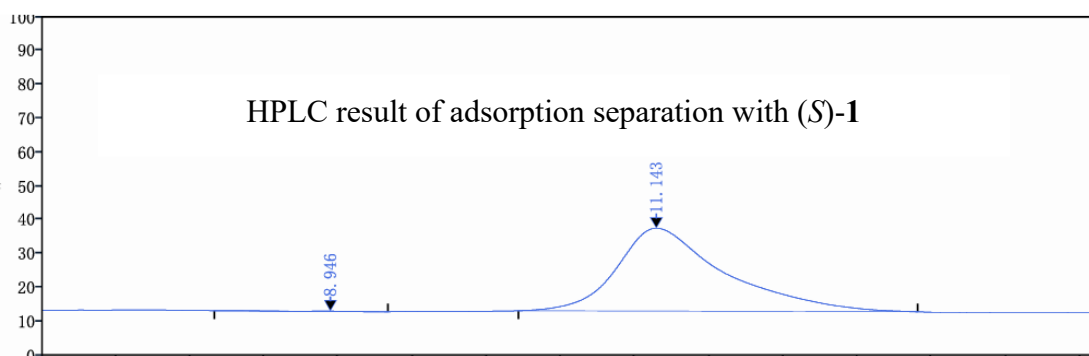
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	12.503	6.70	0.41
2	MM	15.927	1609.88	99.59
The Total			1616.58	



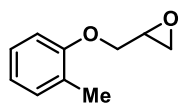
Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 220 nm; $t_R = 8.725$ min, $t_R = 10.895$ min.



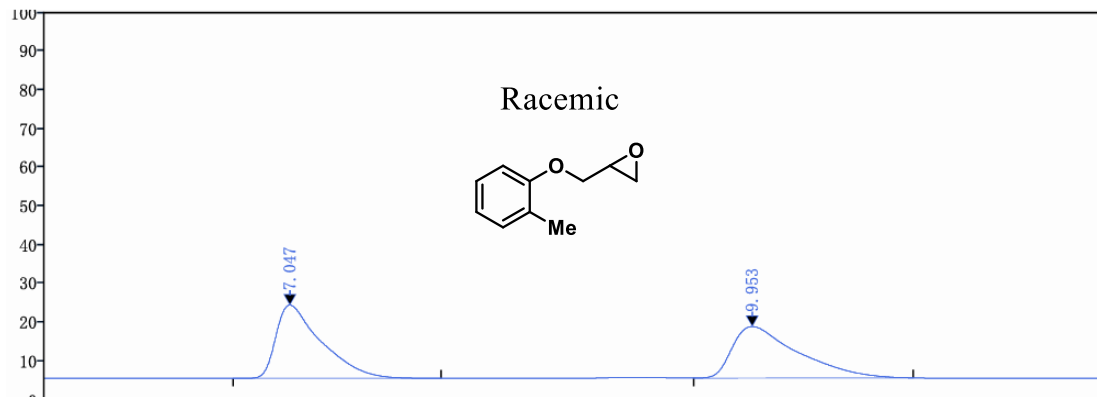
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.725	1467.44	49.84
2	MM	10.895	1476.88	50.16
The Total			2944.32	



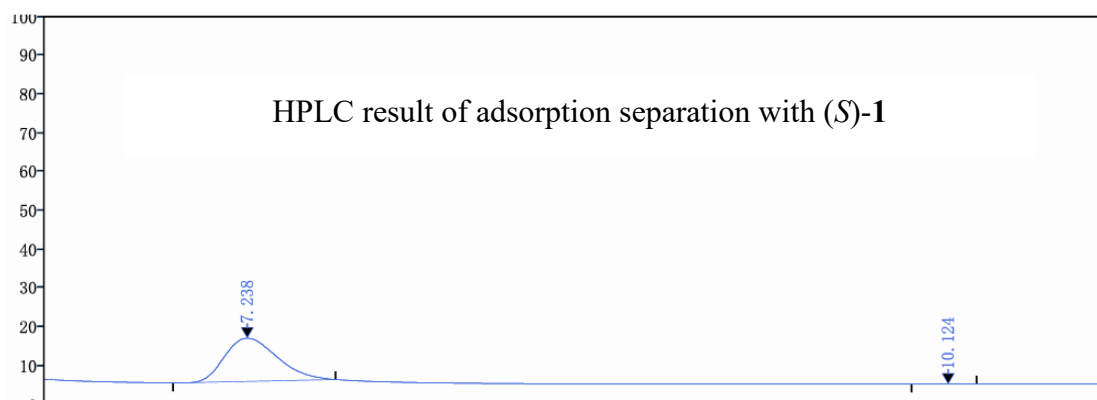
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.946	0.13	0.02
2	MM	11.143	841.57	99.98
The Total			841.70	



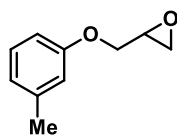
Chiral HPLC analysis: Daicel Chiralce IA: hexane/*i*-PrOH =98/2; flow rate =1.0 mL/min; 220 nm; $t_R = 7.047$ min, $t_R = 9.953$ min.



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	7.047	39843.52	49.52
2	MM	9.953	40612.18	50.48
The Total			80455.70	

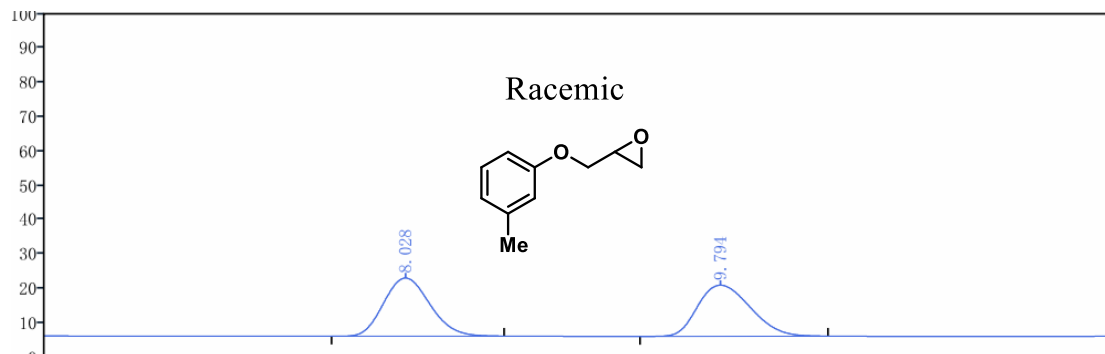


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	7.238	551.63	100.00
2	MM	10.124	0.00	0.00
The Total			551.63	

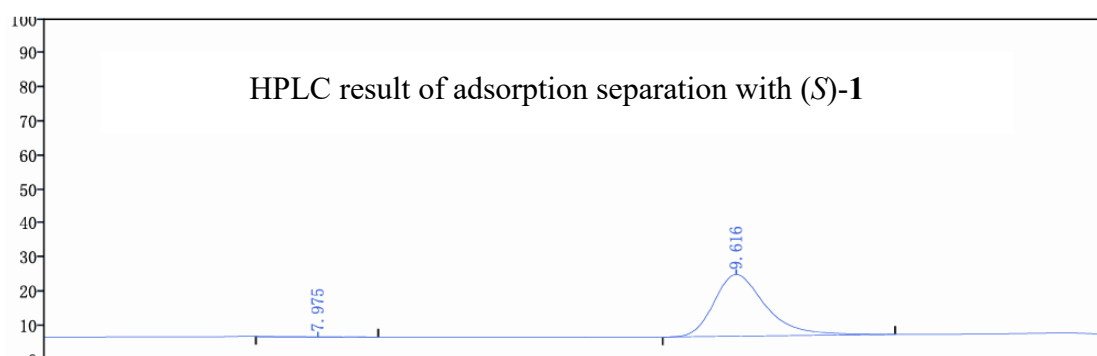


Chiral HPLC analysis: Daicel Chiralce AS: hexane/*i*-PrOH =98/2; flow rate =1.0

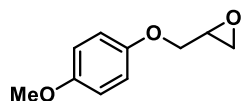
mL/min; 220 nm; t_R =8.028 min, t_R = 9.794 min.



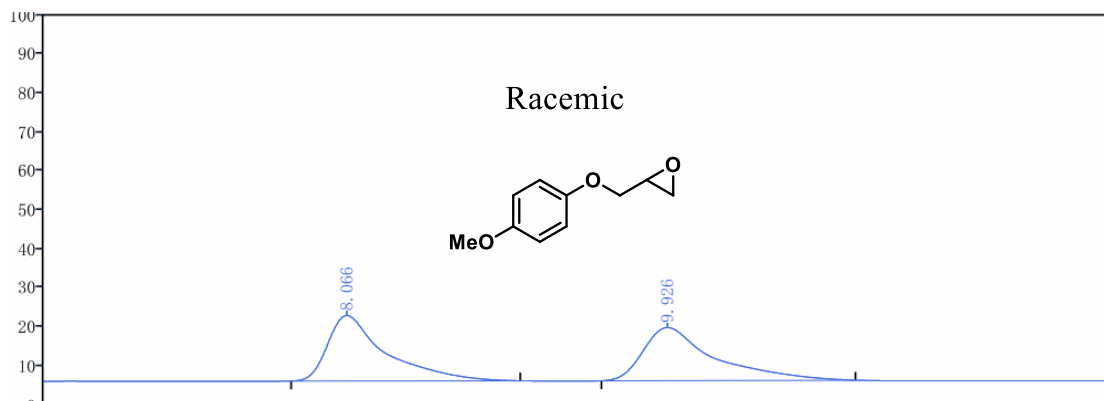
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.028	9378.66	49.98
2	MM	9.794	9384.59	50.02
The Total			18763.25	



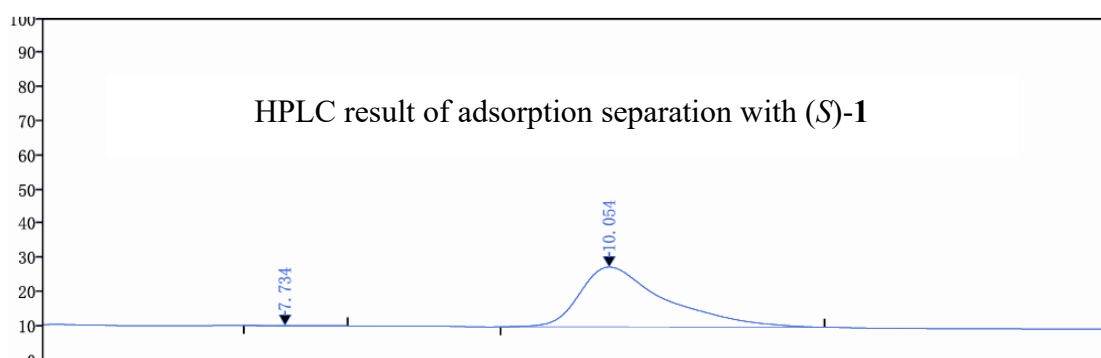
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	7.975	0.51	0.09
2	MM	9.616	575.11	99.91
The Total			575.62	



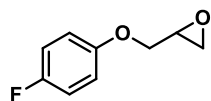
Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =90/10; flow rate =1.0 mL/min; 254 nm; $t_R = 8.066$ min, $t_R = 9.926$ min.



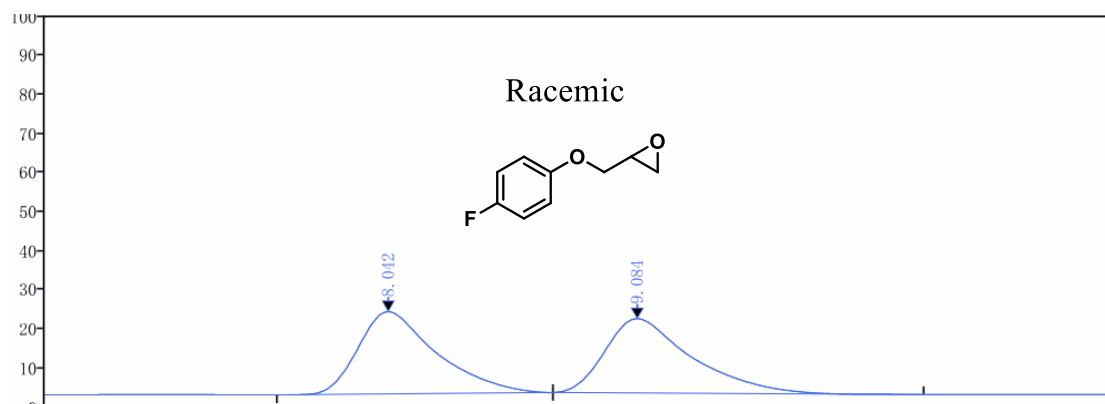
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.066	1488.37	50.27
2	MM	9.926	1472.50	49.73
The Total			2960.87	



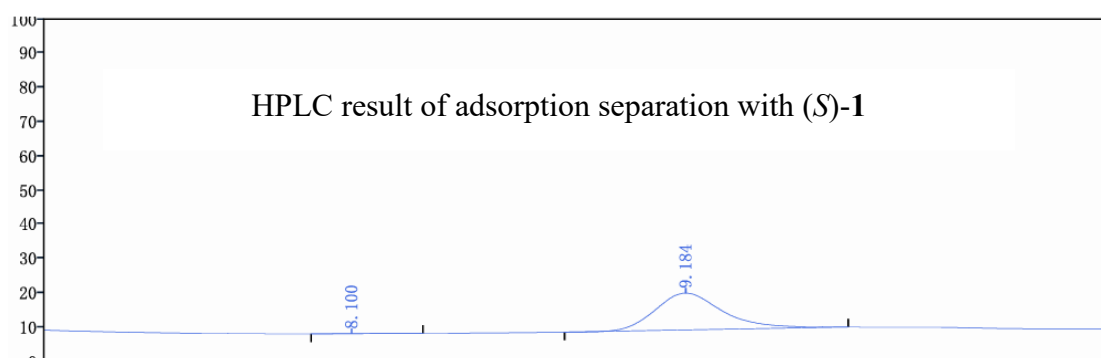
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	7.734	0.56	0.10
2	MM	10.054	588.90	99.90
The Total			589.46	



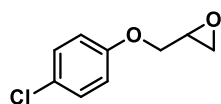
Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =98/2; flow rate =1.0 mL/min; 220 nm; $t_R = 8.042$ min, $t_R = 9.084$ min.



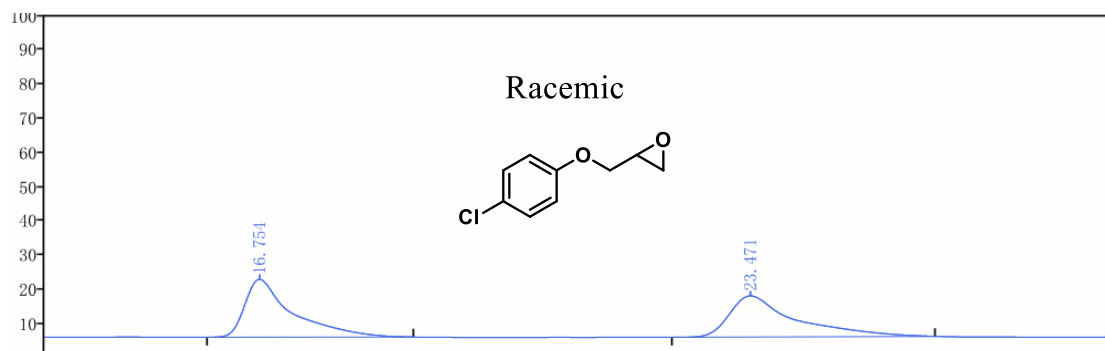
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.042	14870.59	49.98
2	MM	9.084	14883.19	50.02
The Total			29753.78	



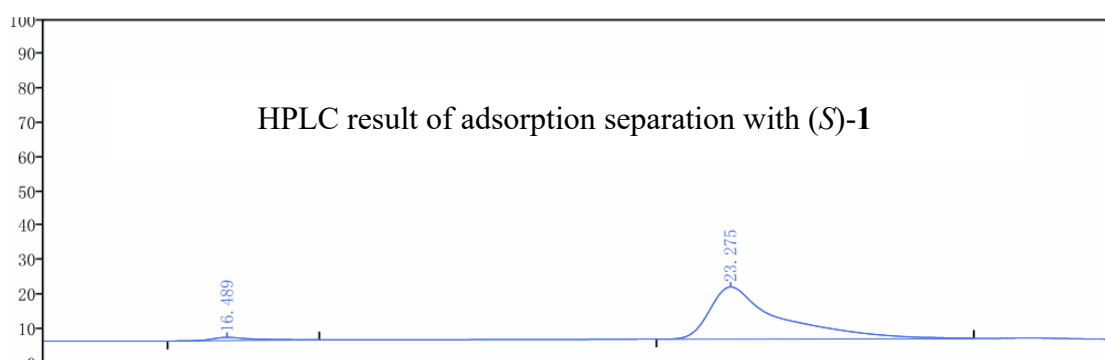
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.100	0.39	0.06
2	MM	9.184	694.13	99.94
The Total			694.52	



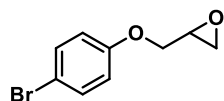
Chiral HPLC analysis: Daicel Chiralce OJ: hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 220 nm; $t_R = 16.754$ min, $t_R = 23.471$ min.



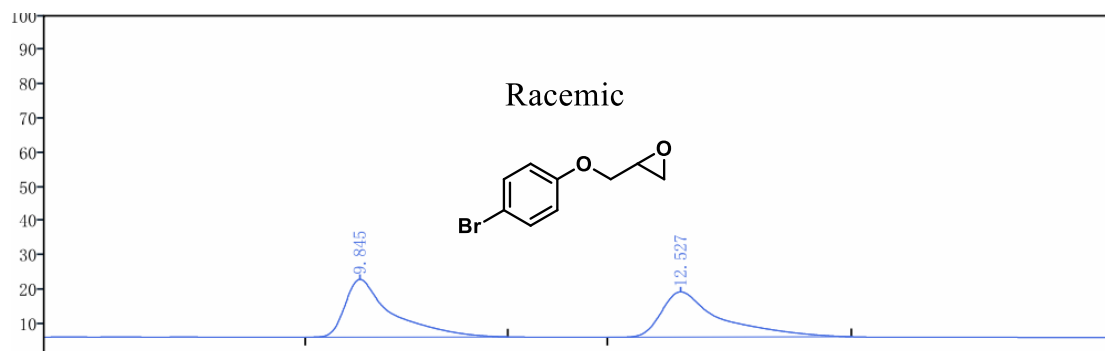
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	16.754	975.50	50.36
2	MM	23.471	961.72	49.64
The Total			1937.22	



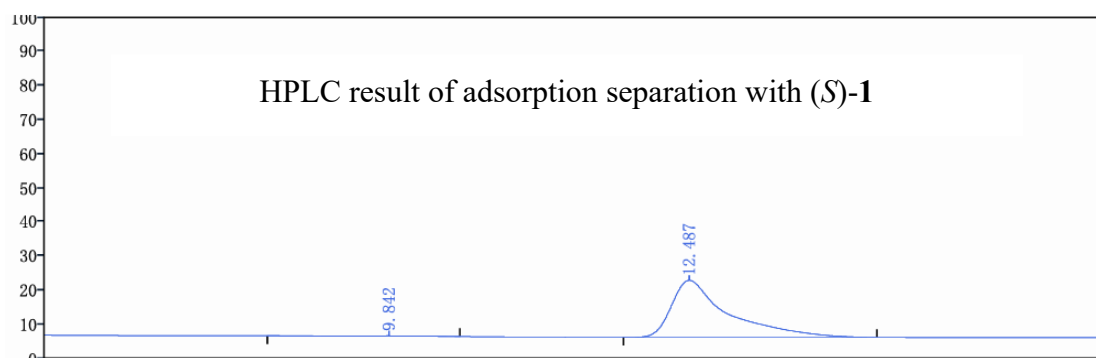
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	16.489	0.69	0.12
2	MM	23.275	580.15	99.88
The Total			580.84	



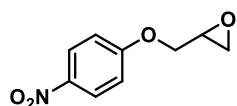
Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =90/10; flow rate =1.0 mL/min; 230 nm; $t_R = 9.845$ min, $t_R = 12.527$ min.



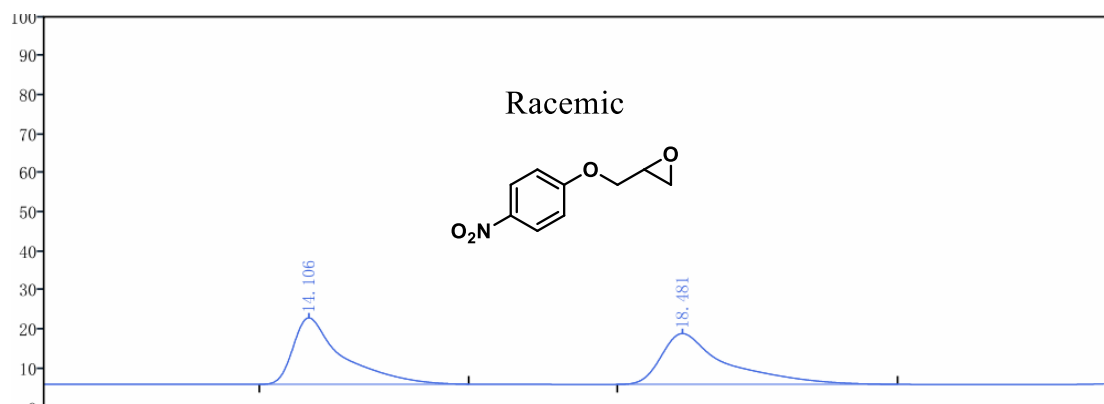
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.845	1023.62	50.16
2	MM	12.527	1016.95	49.84
The Total			2040.57	



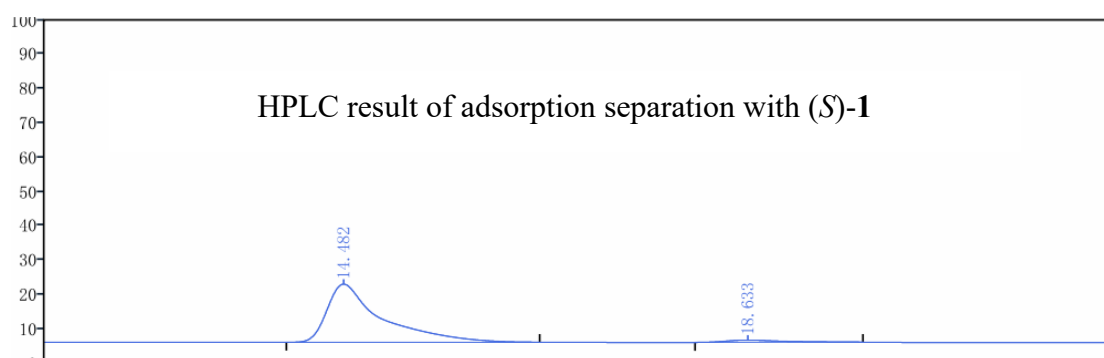
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.842	5.30	0.57
2	MM	12.487	920.68	99.43
The Total			925.98	



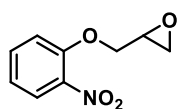
Chiral HPLC analysis: Daicel Chiralce AD: hexane/*i*-PrOH =90/10; flow rate =1.0 mL/min; 218 nm; $t_R = 14.106$ min, $t_R = 18.481$ min.



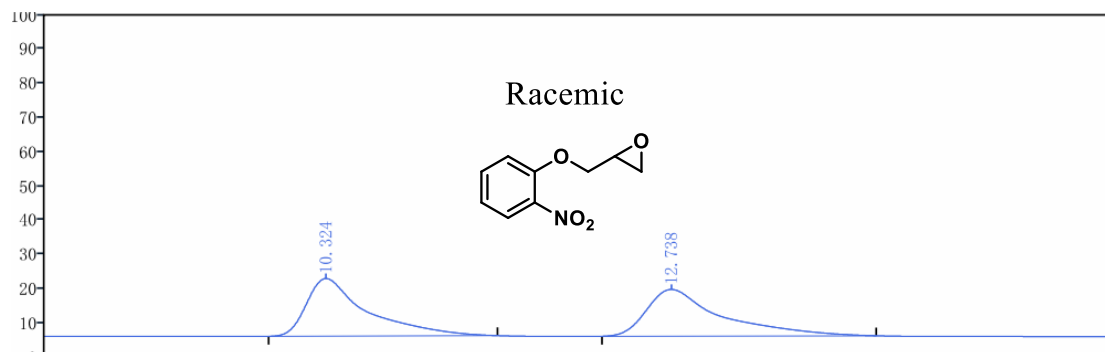
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	14.106	2741.11	49.83
2	MM	18.481	2759.56	50.17
The Total			5500.67	



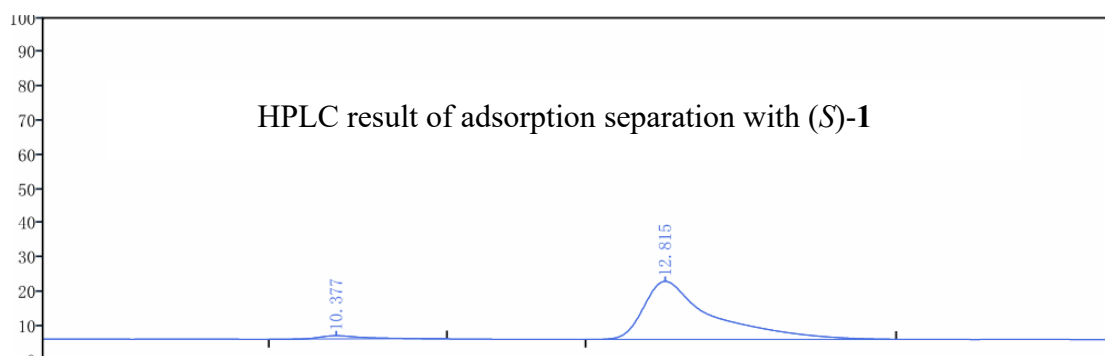
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	14.482	1205.24	96.57
2	MM	18.633	42.87	3.43
The Total			1248.11	



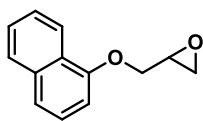
Chiral HPLC analysis: Daicel Chiralce IA: hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 220 nm; $t_R = 10.324$ min, $t_R = 12.738$ min.



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	10.324	1754.72	50.01
2	MM	12.738	1753.70	49.99
The Total			3508.42	

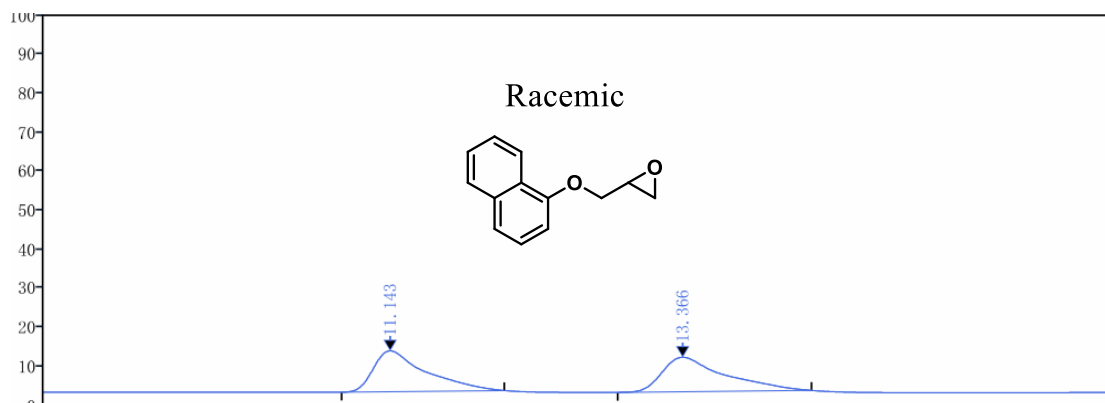


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	10.377	29.70	3.01
2	MM	12.815	955.96	96.99
The Total			985.66	

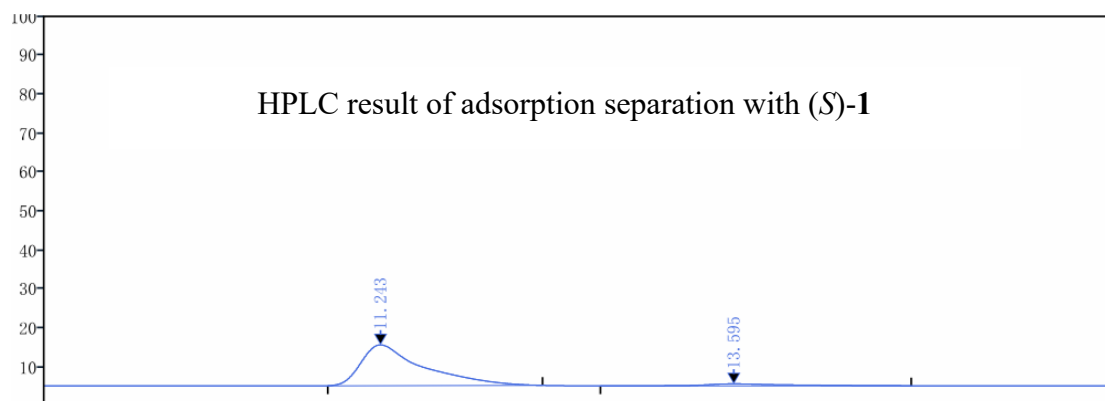


Chiral HPLC analysis: Daicel Chiralce AS: hexane/*i*-PrOH =98/2; flow rate =1.0

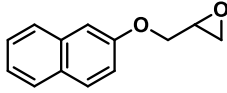
mL/min; 220 nm; $t_R = 11.143$ min, $t_R = 13.366$ min.



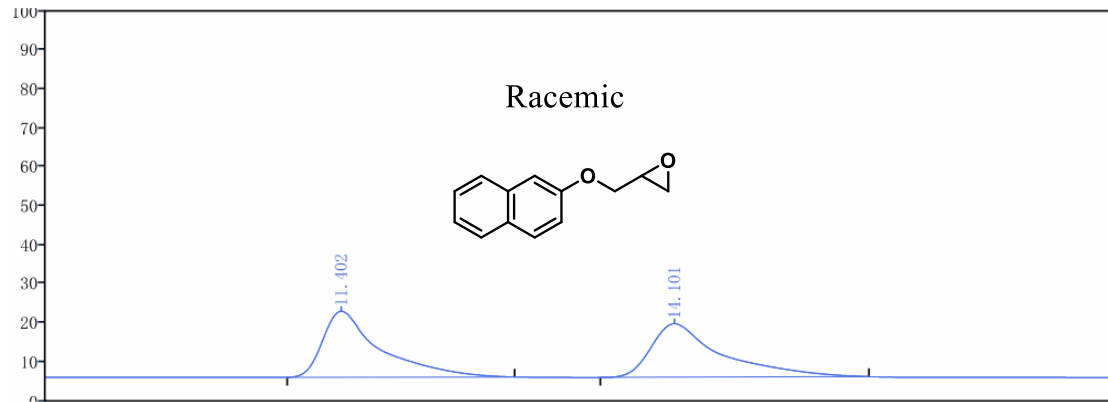
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	11.143	2028.51	49.98
2	MM	13.366	2030.02	50.02
The Total			4058.53	



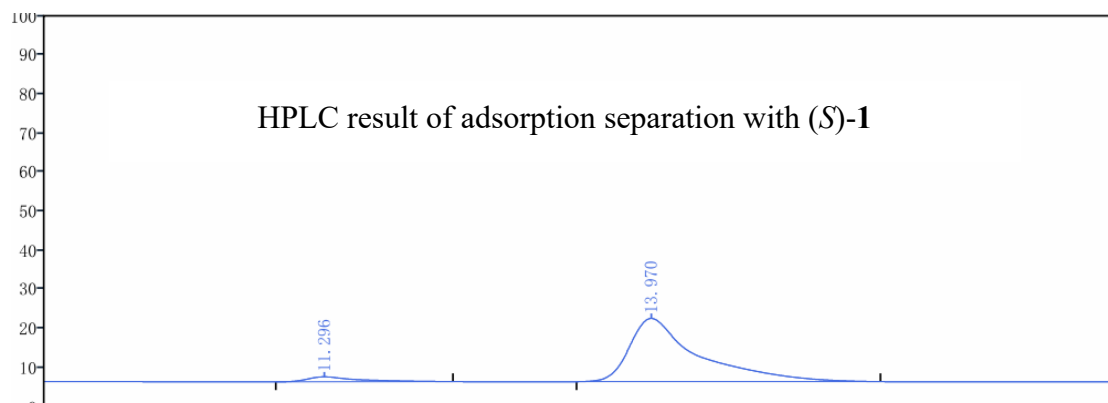
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	11.243	1264.28	94.07
2	MM	13.595	79.66	5.93
The Total			1343.94	



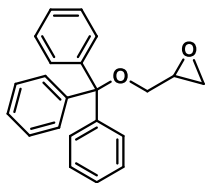
Chiral HPLC analysis: Daicel Chiralce OJ: hexane/*i*-PrOH =70/30; flow rate =1.0 mL/min; 254 nm; $t_R = 11.402$ min, $t_R = 14.101$ min.



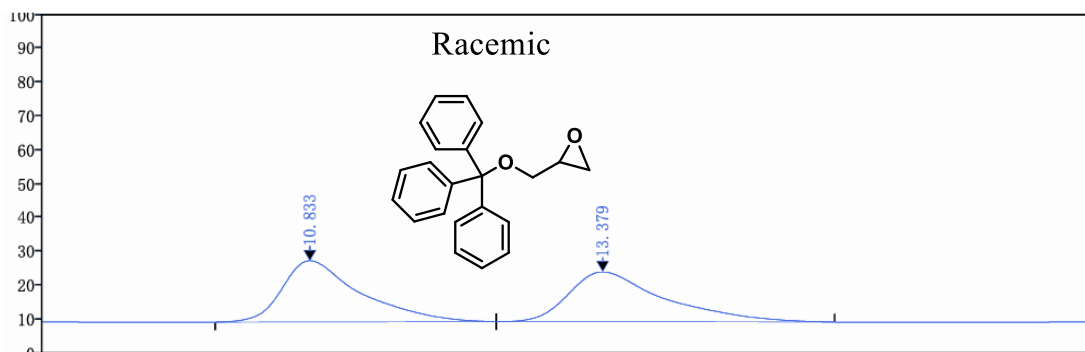
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	11.402	1625.14	50.13
2	MM	14.101	1616.72	49.87
The Total			3241.86	



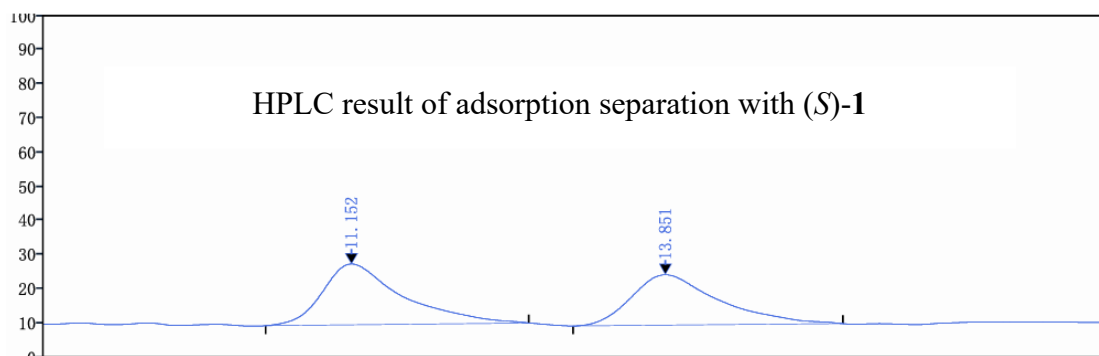
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	11.296	64.83	5.47
2	MM	13.970	1121.42	94.53
The Total			1186.25	



Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 254 nm; $t_R = 10.833$ min, $t_R = 13.379$ min.

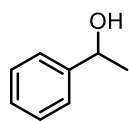


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	10.833	2391.73	50.19
2	MM	13.379	2373.58	49.81
The Total			4765.31	



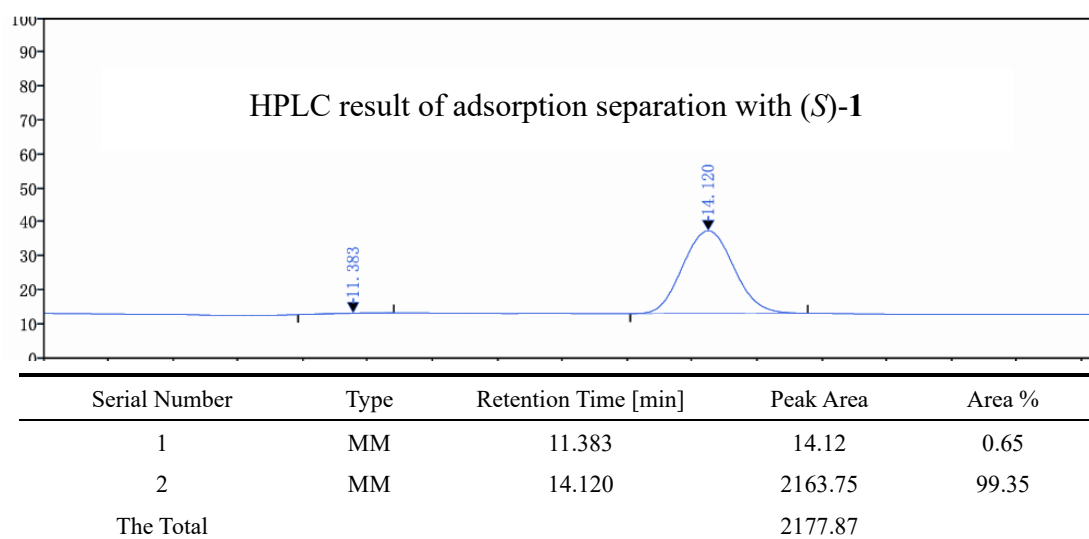
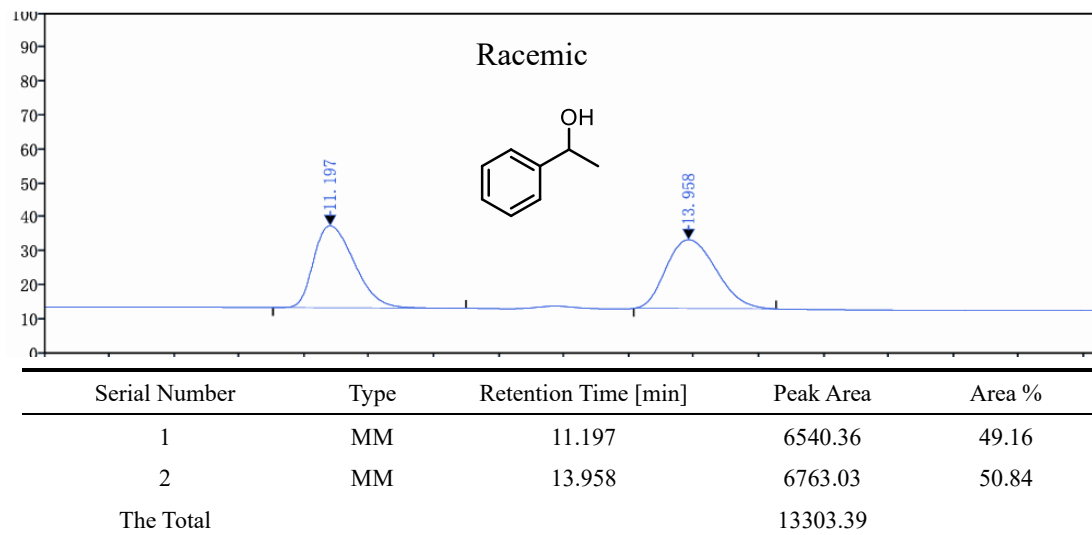
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	11.152	185.78	52.47
2	MM	13.851	168.30	47.53
The Total			354.08	

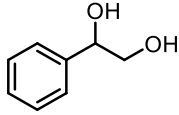
11. Figure S9. HPLC spectra of 1-phenylethanol, phenylethediol, and sec-butylamine



Chiral HPLC analysis: Daicel Chiralce OD-H: hexane/*i*-PrOH =97/3; flow rate

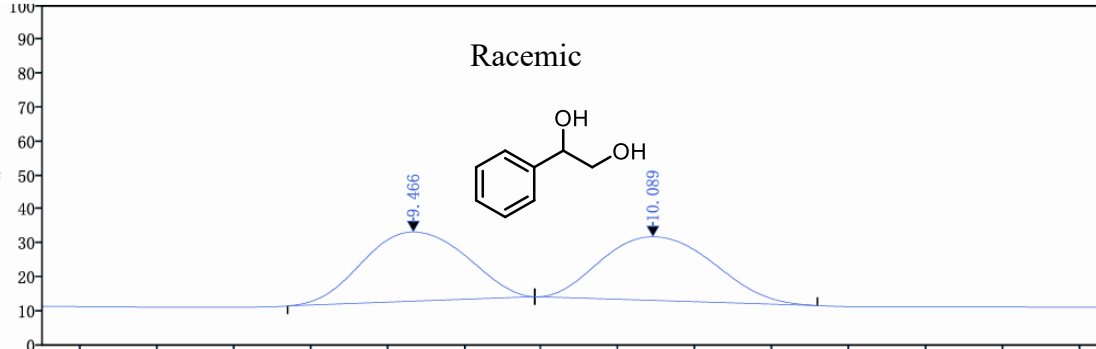
=1.0 mL/min; 220 nm; $t_R = 11.197$ min, $t_R = 13.958$ min.



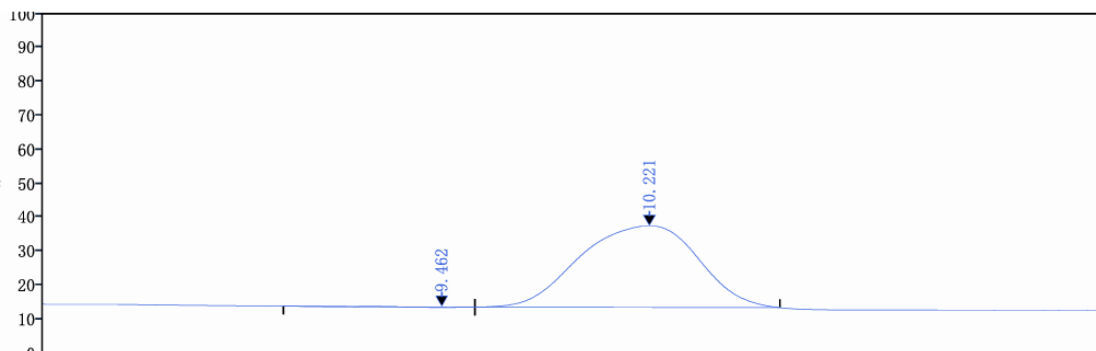


Chiral HPLC analysis: Daicel Chiralce IC: hexane/*i*-PrOH =90/10; flow rate

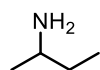
=1.0 mL/min; 220 nm; $t_R = 9.466$ min, $t_R = 10.089$ min.



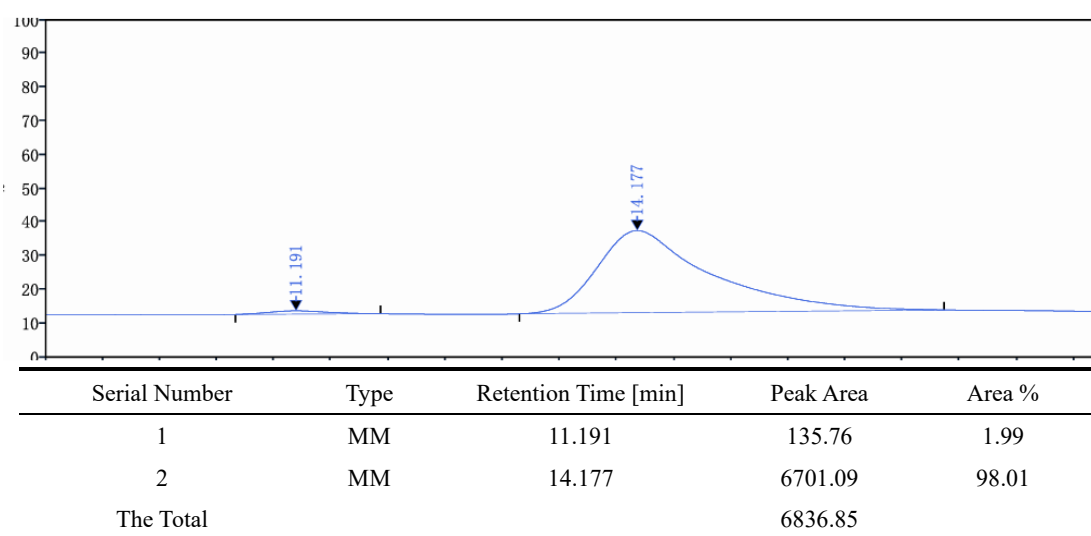
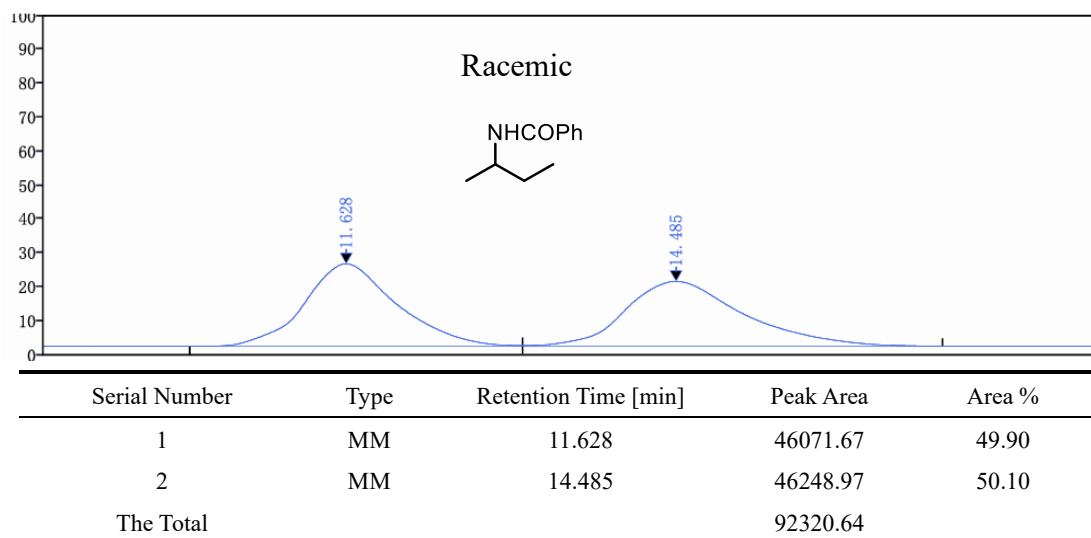
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.466	3799.15	49.98
2	MM	10.089	3802.28	50.02
The Total			7601.43	



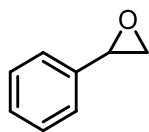
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.462	20.31	0.29
2	MM	10.221	7059.80	99.71
The Total			7080.11	



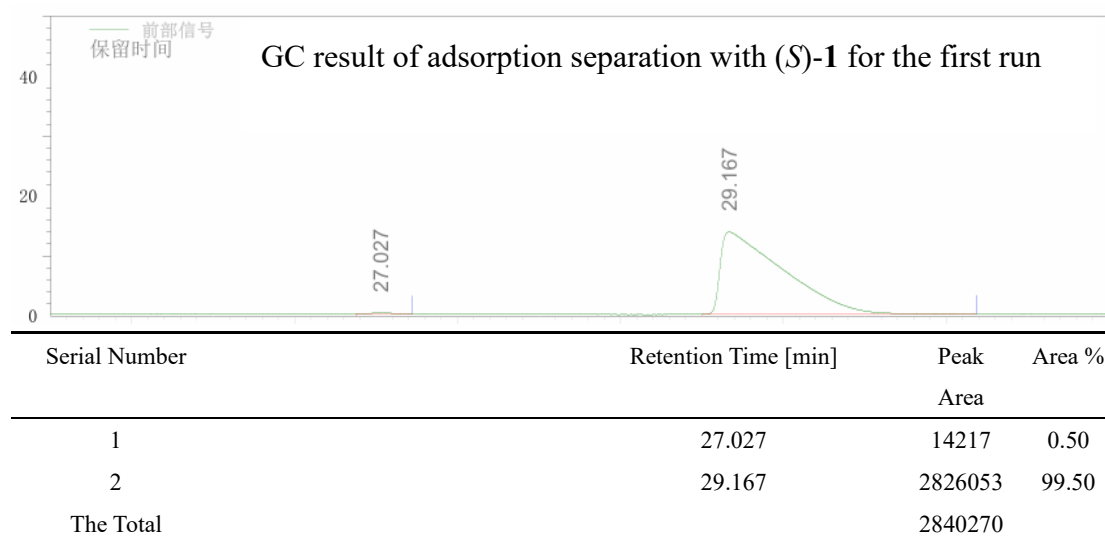
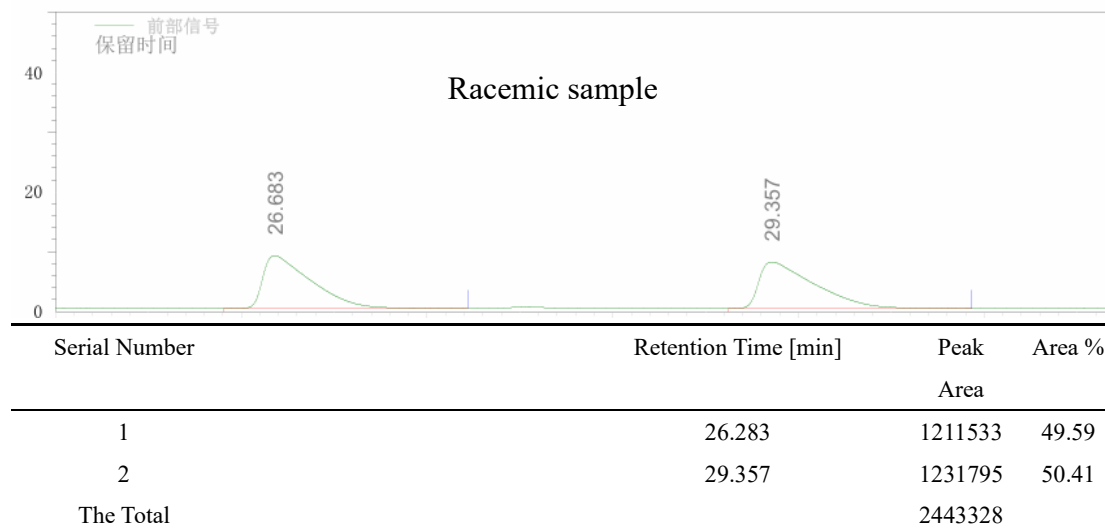
Chiral HPLC analysis: Daicel Chiralce OD-H: hexane/*i*-PrOH =94/6; flow rate =0.8 mL/min; 220 nm; $t_R = 11.628$ min, $t_R = 14.485$ min. (acylated with benzoyl chloride)

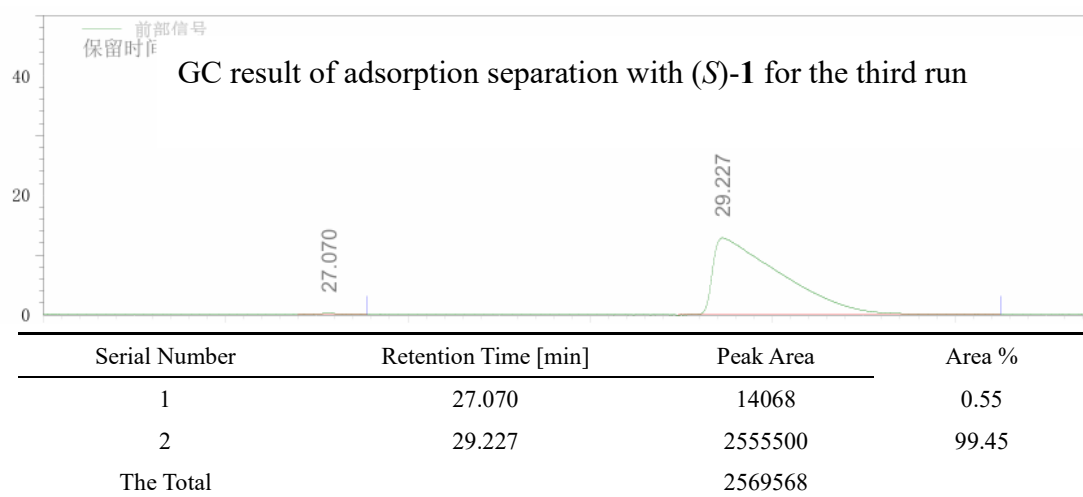
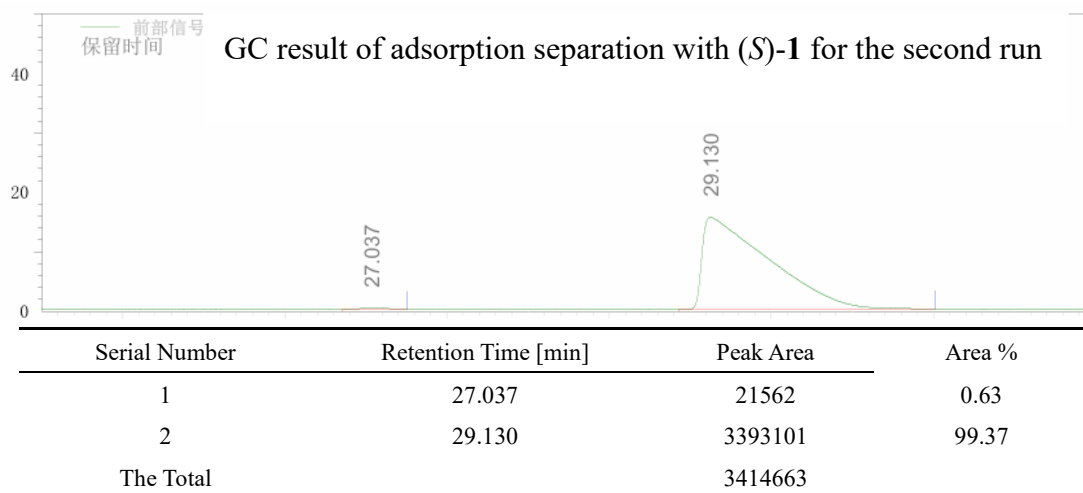


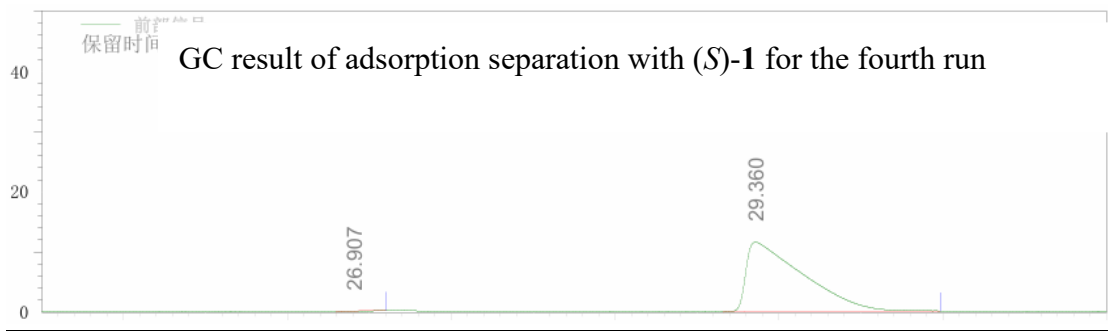
12. Figure S10. GC spectra of SO during the cyclic enantioseparation



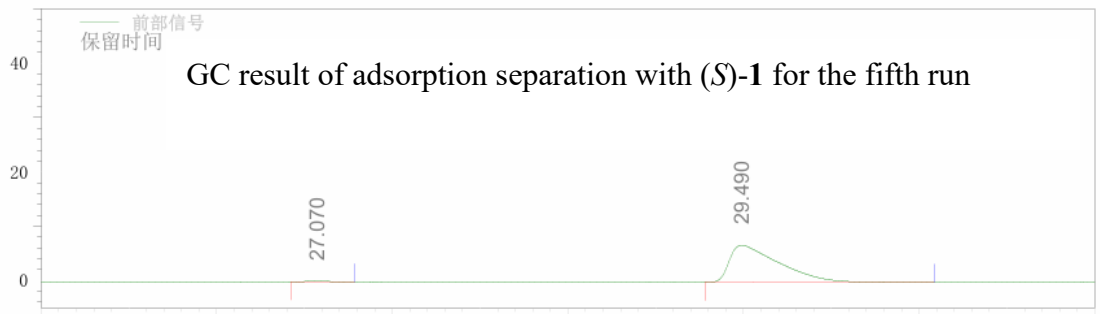
GC analysis: Chirasil-Dex CB; N₂, flow rate = 1.4906 mL/min, 85°C, t_R = 26.683 min, t_R = 29.357 min.







Serial Number	Retention Time [min]	Peak Area	Area %
1	26.907	5382	0.25
2	29.360	2120812	99.75
The Total		2126194	



Serial Number	Retention Time [min]	Peak Area	Area %
1	27.070	8895	0.85
2	29.490	1038046	99.15
The Total		1046941	