Electronic Supplementary Material (ESI) for Dalton Transactions.

Construction of A Chiral Zinc-Camphorate Framework for Enantioselective Separation

Table of Content

1. Materials and general procedures.

All of the chemicals are commercially available and used without any further purification. Single-crystal XRD data for *D*-**1** were collected several times at 100 K at NFPS (Shanghai) synchrotron radiation on BL17B beamline using λ = 0.67042 Å, and the obtained dataset was indexed, integrated and scaled using the APEX4 program. The structure of *D*-**1** was solved by the direct methods with SHELXS-2018 and refined with SHELXL-2018 using *OLEX 2-1.2*. All the hydrogen atoms attached to the ligand were placed in calculated positions and refined using a riding model. Contributions to scattering due to these highly disordered guest molecules in *D*-**1** were removed using the SQUEEZE subroutine of the PLATON software package. The structure was then refined again using the resulting new HKL file. *D*-**1** can be best formulated as $[Zn_2(D\text{-Cam})_2(DPTPA)\cdot 2H_2O]\cdot 2DMF\cdot 2MeOH]$, on the basis of single-crystal diffraction, IR spectra, UV-vis spectra, FL 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 Tables S2. CCDC number of *D*-**1** is 2167425, 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. Thermogravimetric analysis of *D*-**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 Kα radiation. The simulated powder pattern was calculated using Mercury based on single crystal diffraction data of *D*-**1**. The date of dye absorption experiment was recorded on an Agilent Technologies carry UV/Vis Spectrometer. The IR (KBr pellet) spectrum was recorded (400-4000 cm^{-1} region) on a Nicolet Magna 750 FTIR spectrometer. Fluorescence spectra were recorded on a PerkinElmer FL 8500 spectrometer. Analytical high-performance liquid chromatography (HPLC) was performed on an Agilent Technologies 1260 Infinity II with UV detection. Analytical ChiralCel OD-H/OJ-H/AS-H/IC-H/AD-H/IA-H column (4.6 mm × 25 cm) from Daicel were used.

2. Synthesis of *D*-1 and *L*-1.

A mixture of $ZnCl_2$ (2.7 mg, 0.02 mmol), *D*-camphoric acid (*D*-$CamH_2$) (4 mg, 0.02 mmol) and DPTPA co-ligand (6.4 mg, 0.02 mmol) was placed in a glass vial containing DMF (2 mL), MeOH (2 mL), H_2O (1 mL). The vial was sealed tightly and heated at 60°C for two days, colorless block crystals of $[Zn_2(D\text{-Cam})_2(DPTPA)\cdot 2H_2O]\cdot 2DMF\cdot 2MeOH$ (*D*-**1**) were generated. The crystals of *L*-**1** can be obtained under the same synthetic condition as that of *D*-**1** except the *D*-$camH_2$ was instead by *L*-$CamH_2$.

3. Table S1. Crystal data and structure refinement for *D*-1

Identification code	*D*-**1**
Empirical formula	$C_{46}H_{67}N_6O_{16}Zn_2$
Formula weight	1090.79
Temperature (K)	100
Wavelength (Å)	0.67042
Crystal system, space group	Monoclinic, $P2_1$
Unit cell dimensions	a = 8.189(1) Å alpha = 90 deg. b = 20.317(3) Å beta = 98.647(5) deg. c = 17.675(3) Å gamma = 90 deg.
Volume	2907.3(8) $Å^3$
Z, Calculated density	2, 1.246 mg/m^3
Absorption coefficient	0.763 mm^{-1}
F(000)	930
θ range for data collection (º)	1.099 to 23.927
Limiting indices	-9<=h<=9, -24<=k<=24, -21<=l<=21
Reflections collected independent reflections	30140 / 10347 [R(int) = 0.0981]
Completeness to theta Refinement method	98.4 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	10347 / 63 / 494
Goodness-of-fit on F^2	1.066
Final R indices [I>2sigma(I)]	R1 = 0.0879, wR2 = 0.2480
R indices (all data)	R1 = 0.1134, wR2 = 0.2705

4. Table S2. Selected Bond lengths [Å] and angles [º] for *D*-1.

O4-Zn(2)	2.413(7)
O(3)-Zn(2)	2.042(6)
N(4)-Zn(1)	2.088(4)
O(8)-Zn(1)#1	2.028(11)
N(1)-Zn(2)	1.992(4)
O(1)-Zn(1)#2	1.979(10)
O(6)-Zn(2)	2.159(10)
O(11)-Zn(2)	2.138(12)
O(12)-Zn(1)	2.201(13)
O(5)-Zn(2)	2.115(10)
O2-Zn(1) #2	2.429(10)
N(4)-Zn(1)-O(12)	99.2(4)
N(4)-Zn(1)-O(2)#4	155.9(4)
O(8)#3-Zn(1)-N(4)	104.1(3)
O(8)#3-Zn(1)-O(12)	99.0(5)
O(8)#3-Zn(1)-O(2)#4	99.1(4)
O(1)#4-Zn(1)-N(4)	101.1(4)
O(1)#4-Zn(1)-O(8)#3	141.4(5)
O(1)#4-Zn(1)-O(12)	105.3(5)
O(1)#4-Zn(1)-O(2)#4	55.4(5)
O(12)-Zn(1)-O(2)#4	83.5(4)
O(3)-Zn(2)-O(4)	56.1(3)
O(3)-Zn(2)-O(6)	149.5(4)
O(3)-Zn(2)-O(11)	97.3(4)
O(3)-Zn(2)-O(5)	97.0(3)
N(1)-Zn(2)-O(4)	93.8(3)
N(1)-Zn(2)-O(3)	99.3(3)
N(1)-Zn(2)-O(6)	99.8(4)
N(1)-Zn(2)-O(11)	95.0(3)
N(1)-Zn(2)-O(5)	163.2(3)
O(6)-Zn(2)-O(4)	99.0(3)
O(11)-Zn(2)-O(4)	153.1(4)
O(11)-Zn(2)-O(6)	104.4(5)
O(5)-Zn(2)-O(4)	91.6(3)
O(5)-Zn(2)-O(6)	63.5(4)
O(5)-Zn(2)-O(11)	87.2(4)

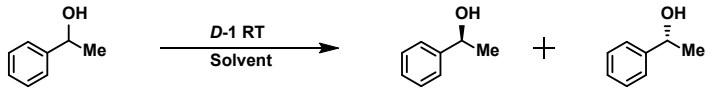
Symmetry transformations used to generate equivalent atoms:

#1 1-x, -1/2+y, -2-z #2 2-x, -1/2+y, -1-z
#3 1-x, 1/2+y, -2-z #4 2-x, 1/2+y, -1-z

5. Experimental procedure for enantioseparation.

A typical procedure for enantioseparation: The solvent-exchanged crystals of *D*-**1** (50 mg) with dry acetone were firstly socked in acetone (2 mL) solution of 1-phenylethanol (1-PE) in different concentrations as the following table for 12 hours (Table S3). After which, the solid crystals were filtered, washed with acetone, and then soaked in fresh acetone to extract the encapsulated chiral 1-PE molecules in *D*-**1**. The optical purity of encapsulated chiral 1-PE molecules was determined by HPLC equipped with chiral column. Other chiral substrates were separated under the identical optimized conditions as entry 2.

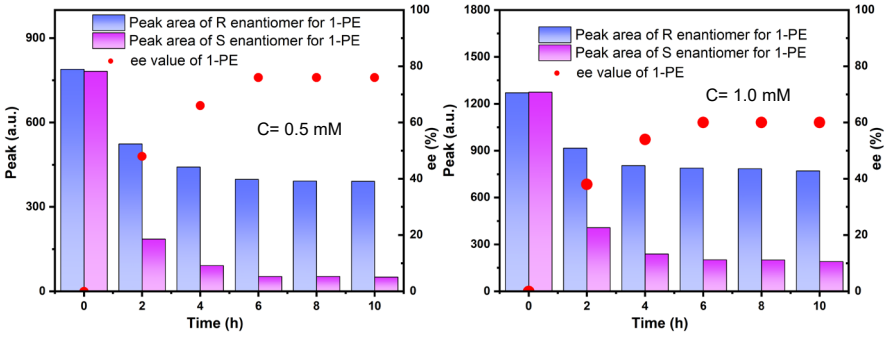
Table S3. Optimization of separation condition of *D*-**1**



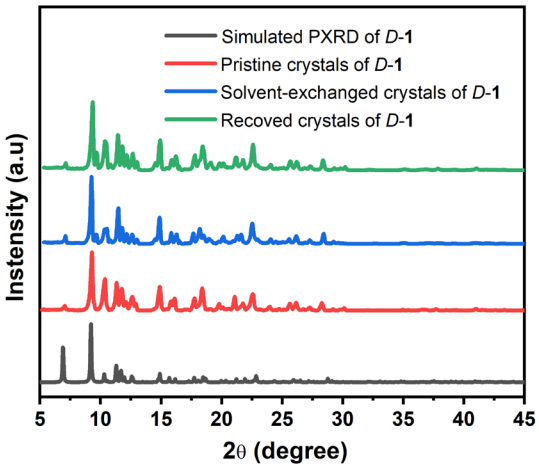
Entry	Host	Solvent	Substrate concentration (mM)	ee(%)
1	*D*-1	Acetone	5	99(*S*)
2	*D*-1	Acetone	10	99(*S*)
3	*D*-1	Acetone	15	95(*S*)
4	*D*-1	Acetone	20	90(*S*)
5	*D*-1	Acetone	30	84(*S*)
6	*D*-1	Acetone	40	74(*S*)
7	*L*-1	Acetone	10	99(*R*)
8	*D*-1	MeOH	10	69(*S*)
9	*D*-1	EtOH	10	50(*S*)
10	*D*-1	THF	10	39(*S*)
11	*D*-1	$CHCl_3$	10	43(*S*)
12	*D*-1	CH_2Cl_2	10	58(*S*)
13	*D*-1	CH_3CN	10	62(*S*)
14	*D*-1	Et_2O	10	34(*S*)

Quantitative Dynamic Adsorption Experiments: The solvent-exchanged crystals of *D*-**1** (50 mg) was immersed in 2 mL of acetone solution of 1-PE at concentration of 0.5 mM

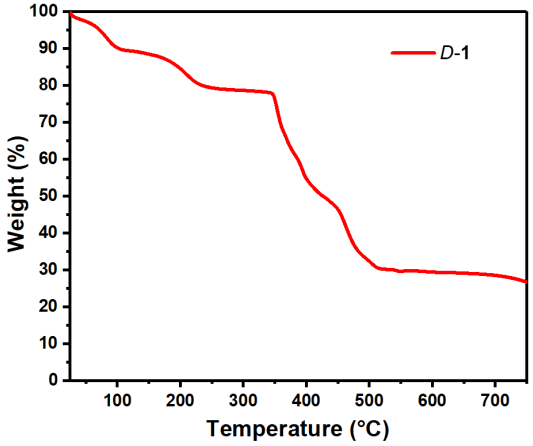
and 1.0 mM, respectively. Then, the *R* and *S* enantiomers of 1-PE in the supernatant were monitored by HPLC in terms of the peak area and ee value with the time. The results indicate that *S* enantiomers of 1-PE was encapsulated preferentially by *D*-**1**.



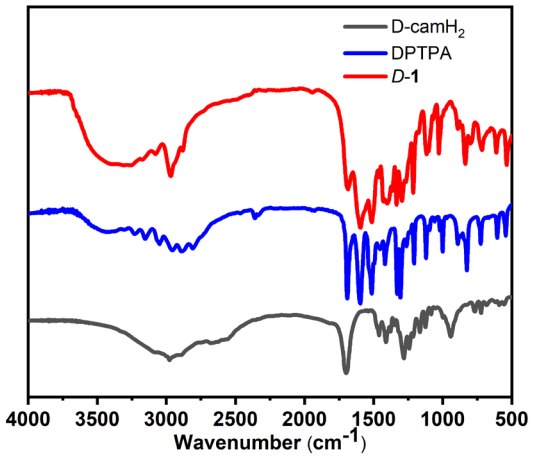
6. Figure S1. PXRD patterns of *D*-1.



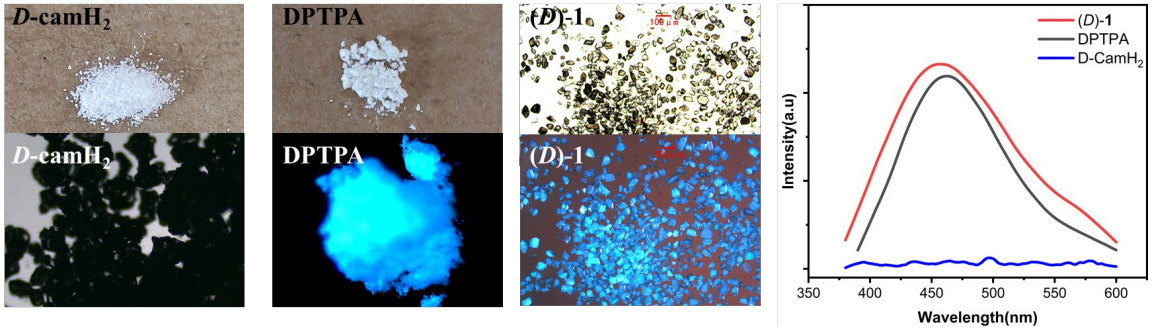
7. Figure S2. TGA curves of *D*-1.



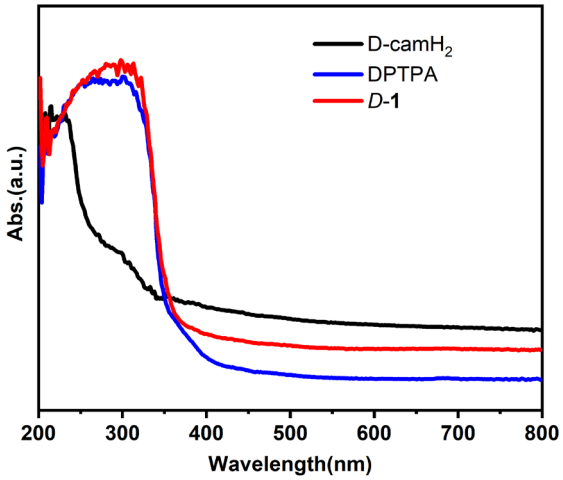
8. Figure S3. IR spectra of *D*-camH$_2$、 DPTPA and *D*-1.



9. Figure S4. View the FL spectra of *D*-camH$_2$、 DPTPA and *D*-1, and their optical photos of under sunlight and UV light, respectively.

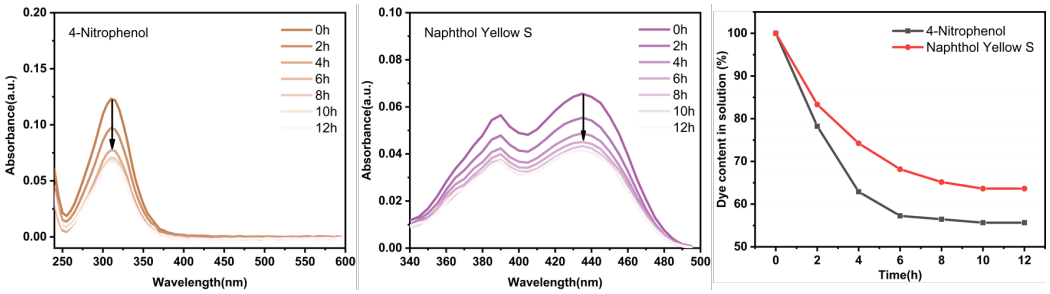


10. Figure S5. UV-vis spectra of *D*-camH$_2$、 DPTPA and *D*-1.

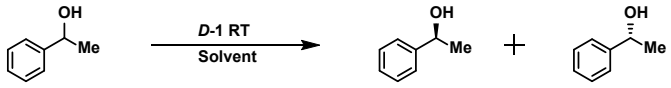


11. Figure S6. The adsorption of *D*-1 to dye molecules.

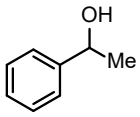
The 20 mg pristine crystals of *D*-**1** were immersed in methanol solution of 10 μmol/L 4-nitrophenol and naphthol yellow S, respectively. Then the dye content in solution was monitored by UV-Vis at different time periods.



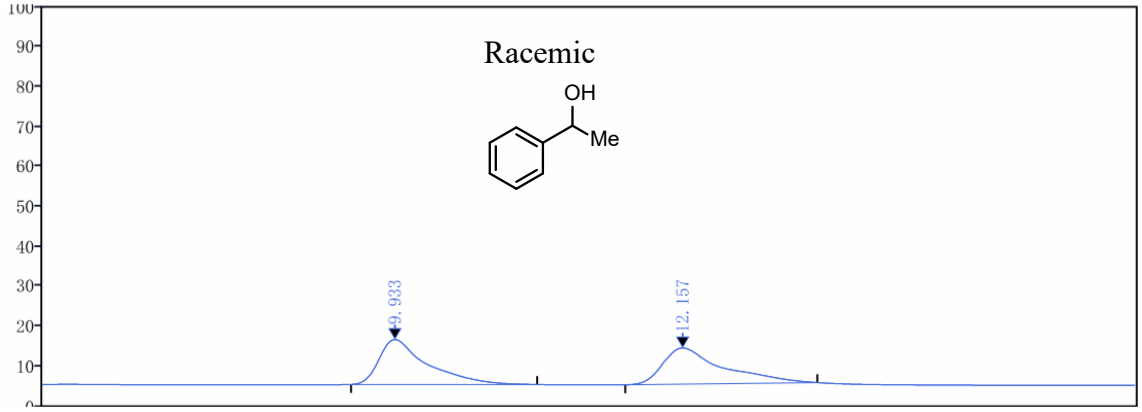
12. Figure S7. HPLC results of 1-phenylethanol resolved by *D*-1 at different conditions (in Table S3).



Entry	Host	Solvent	Substrate concentration (mM)	ee(%)
1	*D*-1	Acetone	5	99(*S*)
2	*D*-1	Acetone	10	99(*S*)
3	*D*-1	Acetone	15	95(*S*)
4	*D*-1	Acetone	20	90(*S*)
5	*D*-1	Acetone	30	84(*S*)
6	*D*-1	Acetone	40	74(*S*)
7	*L*-1	Acetone	10	99(*R*)
8	*D*-1	MeOH	10	69(*S*)
9	*D*-1	EtOH	10	50(*S*)
10	*D*-1	THF	10	39(*S*)
11	*D*-1	$CHCl_3$	10	43(*S*)
12	*D*-1	CH_2Cl_2	10	58(*S*)
13	*D*-1	CH_3CN	10	62(*S*)
14	*D*-1	Et_2O	10	34(*S*)



Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =96/4; flow rate =1.0 mL/min; 220 nm.



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.933	1248.45	49.97
2	MM	12.157	1250.10	50.03
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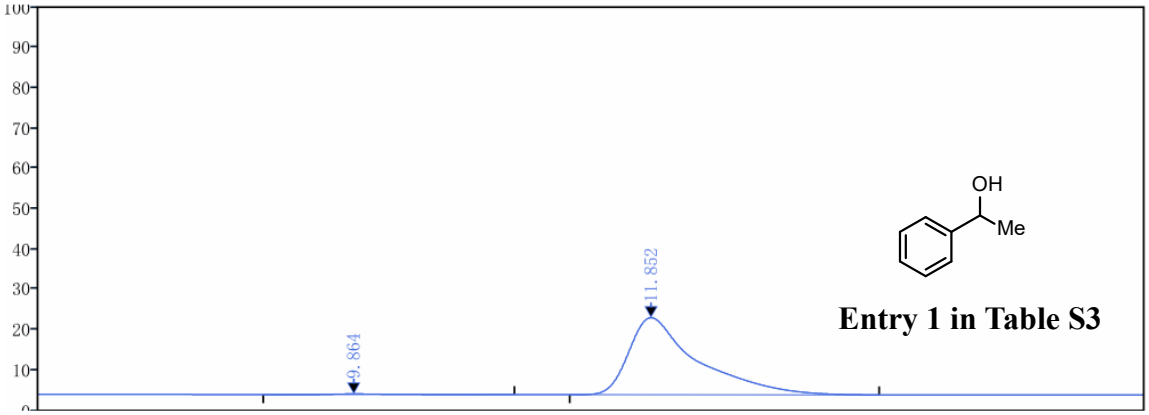
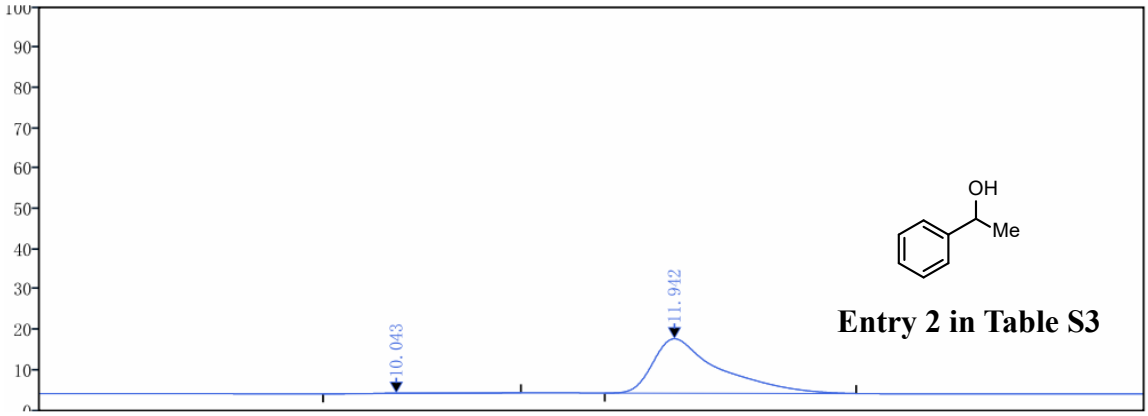


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.864	2.95	0.27
2	MM	11.852	1079.57	99.73
The Total			1082.52	



Serial Number	Type	Retention Time [min]	Peak Area	Area %
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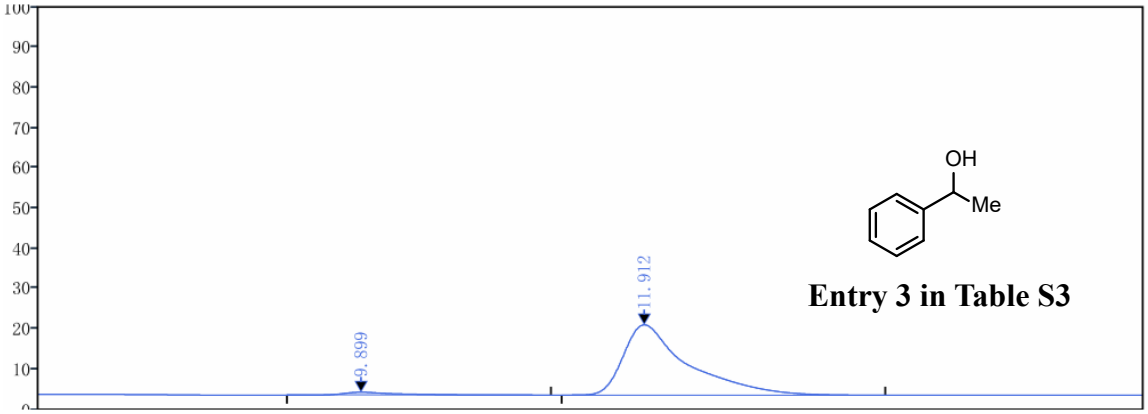
2	BM	11.942	1328.47	99.60
The Total			1333.78	



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MB	9.899	22.46	2.51
2	BB	11.912	871.18	97.49
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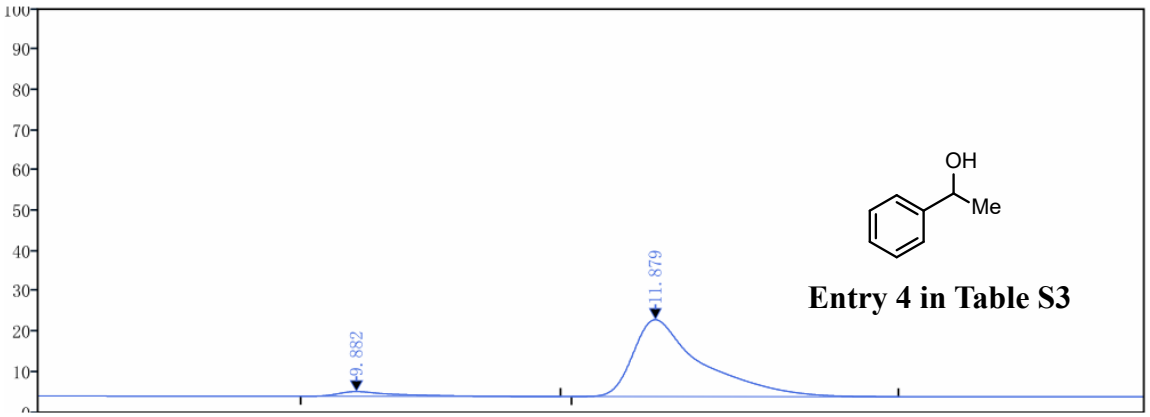

Serial Number	Type	Retention Time [min]	Peak Area	Area %
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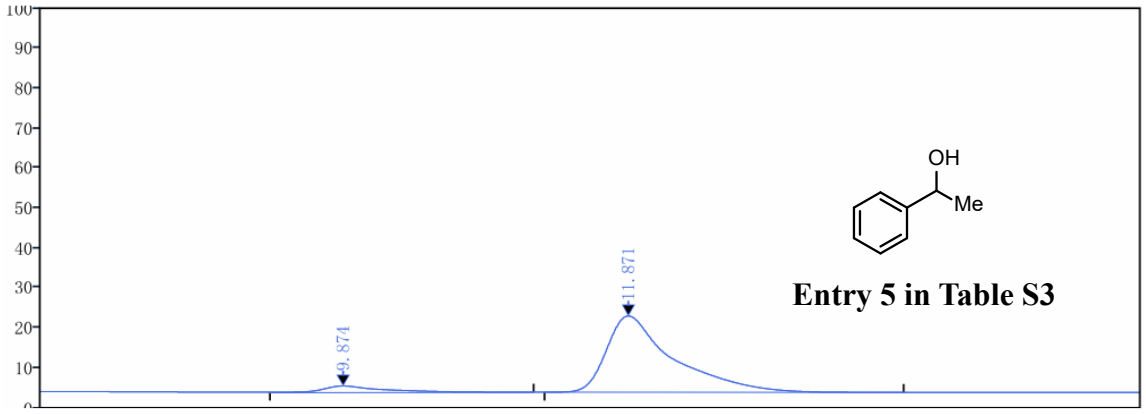
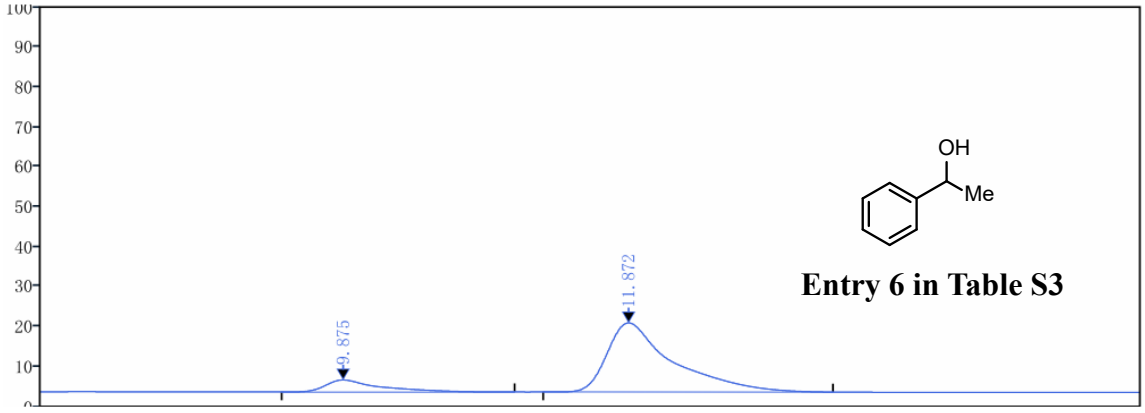
2	BB	11.879	936.49	95.13
The Total			984.46	



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.874	95.68	7.70
2	BM	11.871	1147.22	92.30
The Total			1242.90	



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.875	148.26	13.01
2	MM	11.872	991.31	86.99
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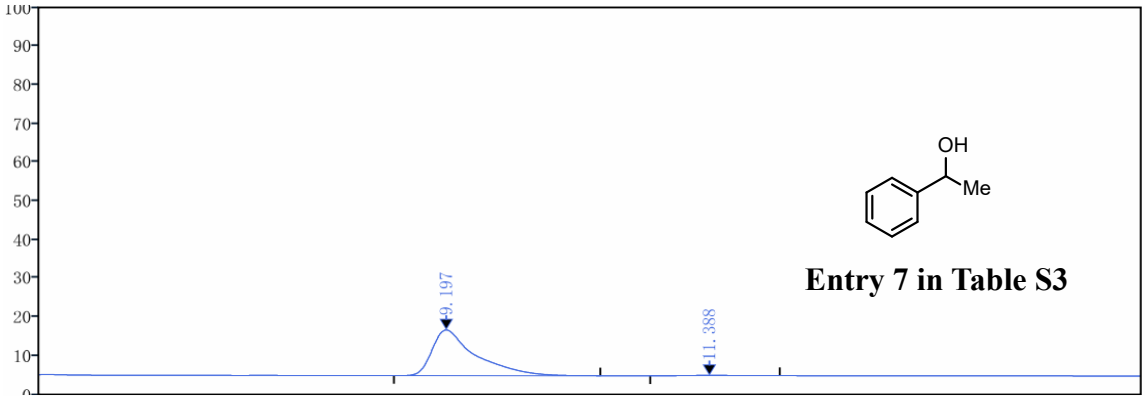

Serial Number	Type	Retention Time [min]	Peak Area	Area %
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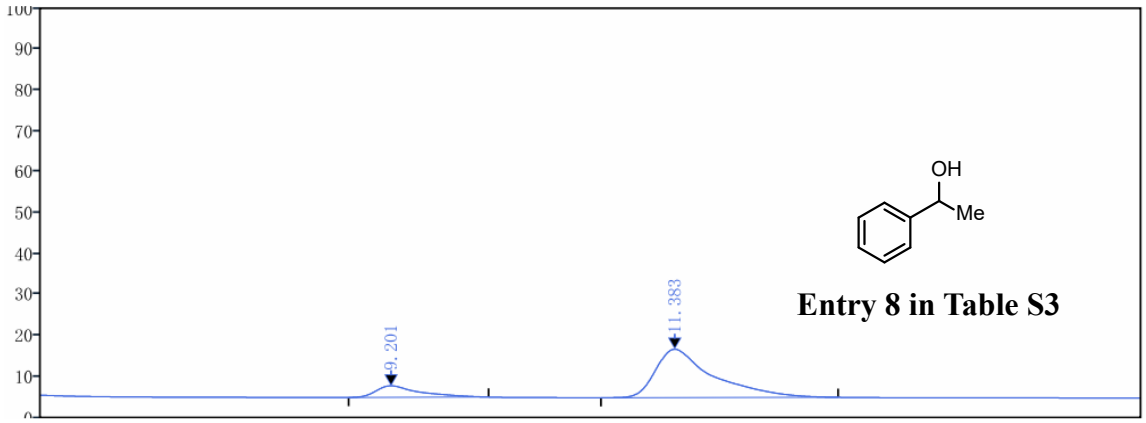

Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.201	248.79	15.22
2	BM	11.383	1386.05	84.78
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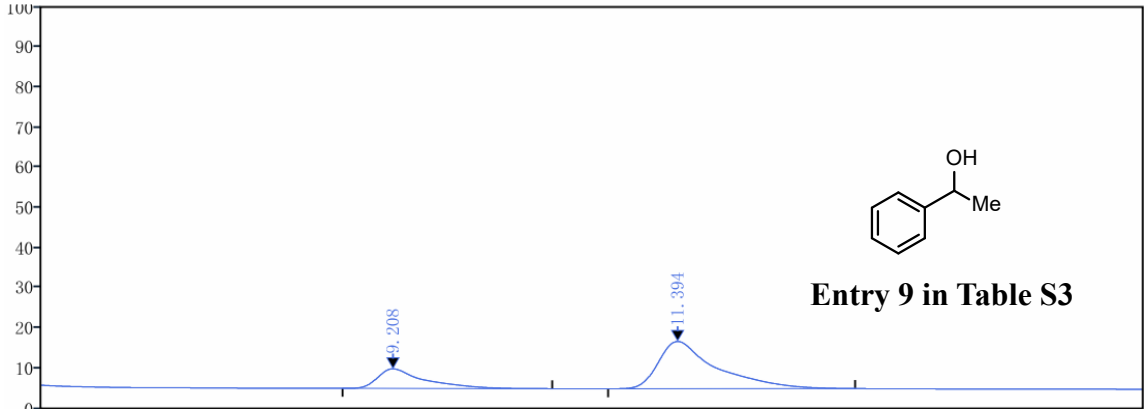

Serial Number	Type	Retention Time [min]	Peak Area	Area %
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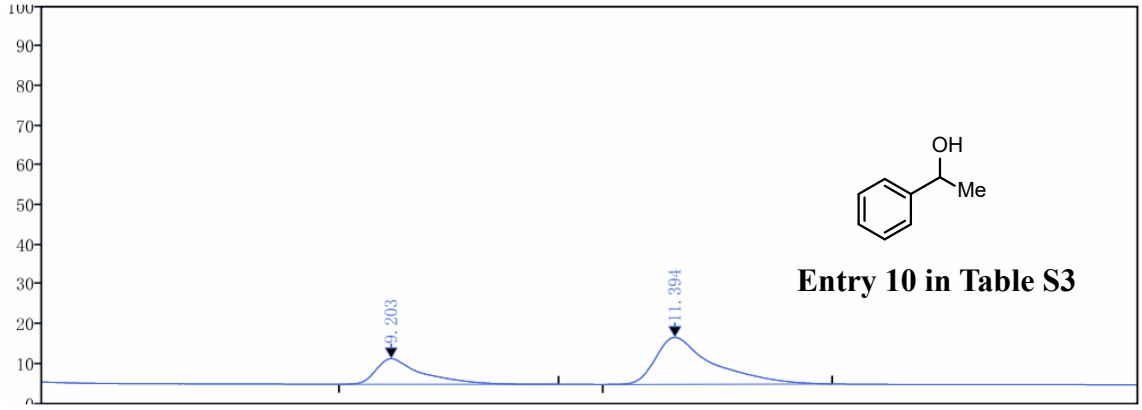

Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BB	9.203	655.23	30.46
2	BM	11.394	1496.14	69.54
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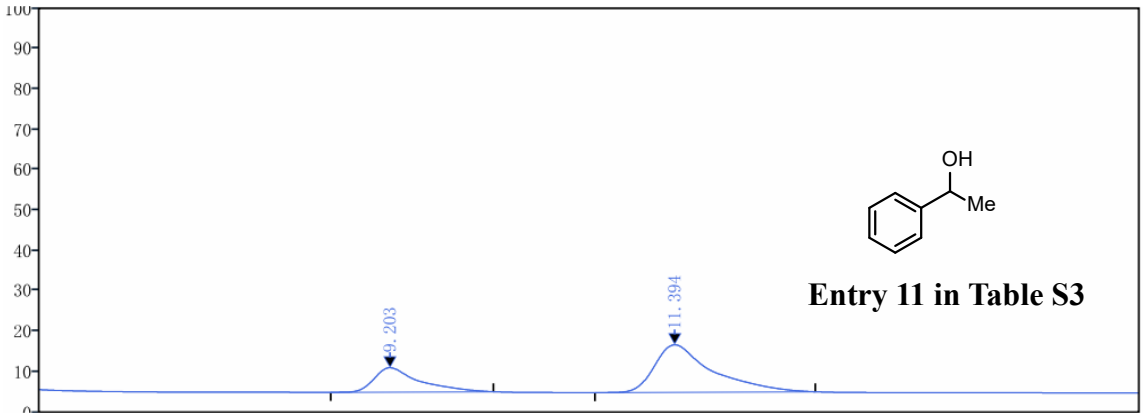

Serial Number	Type	Retention Time [min]	Peak Area	Area %
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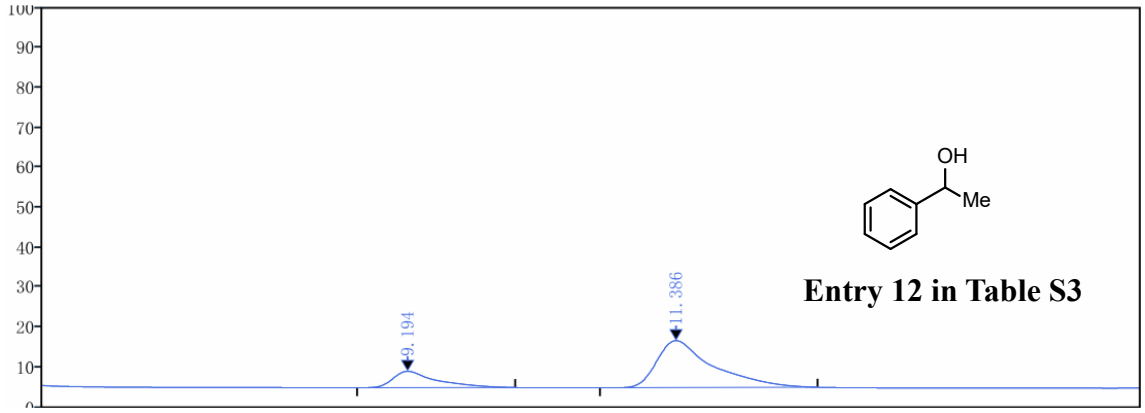

Serial Number	Type	Retention Time [min]	Peak Area	Area %
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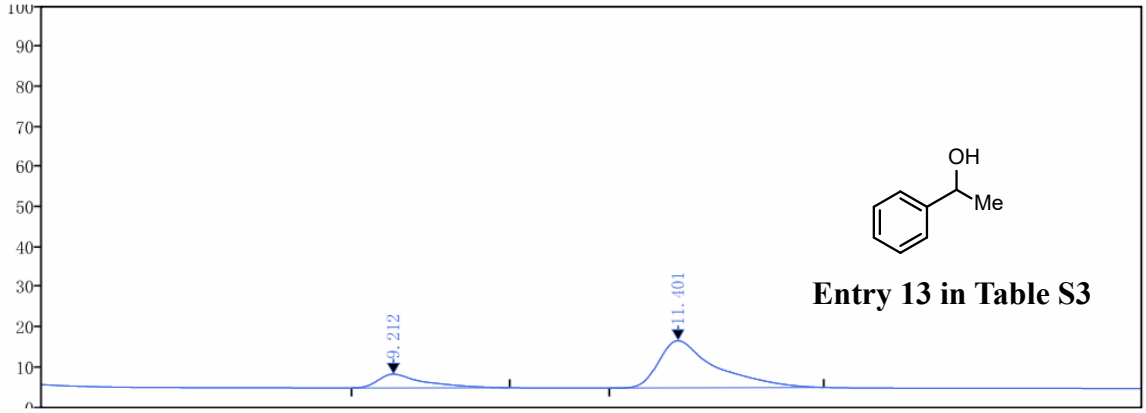
2	BM	11.386	3826.95	78.95
The Total			4847.08	



Serial Number	Type	Retention Time [min]	Peak Area	Area %
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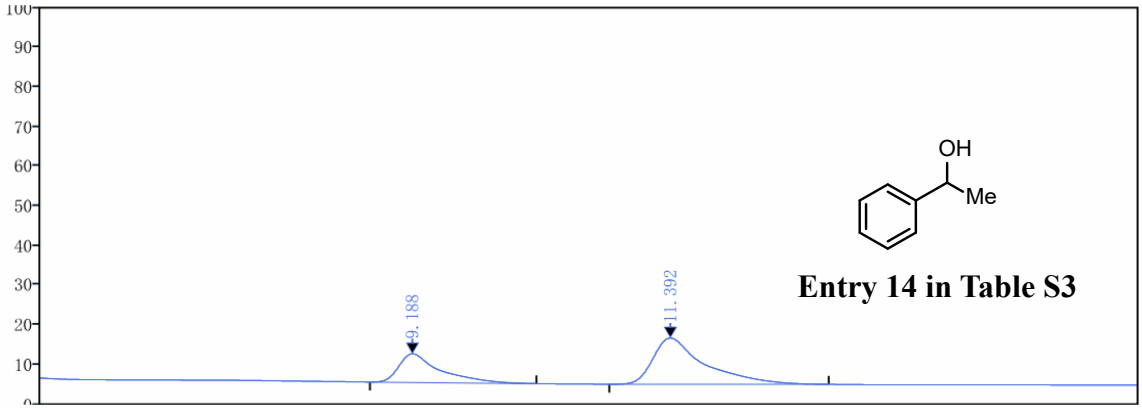

Serial Number	Type	Retention Time [min]	Peak Area	Area %
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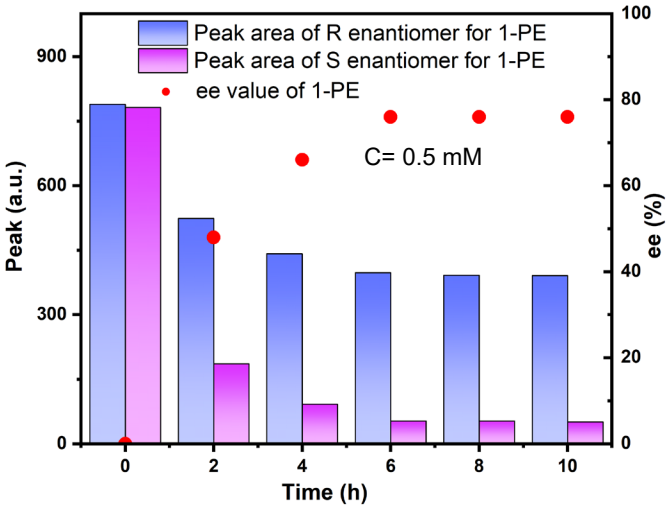
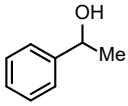
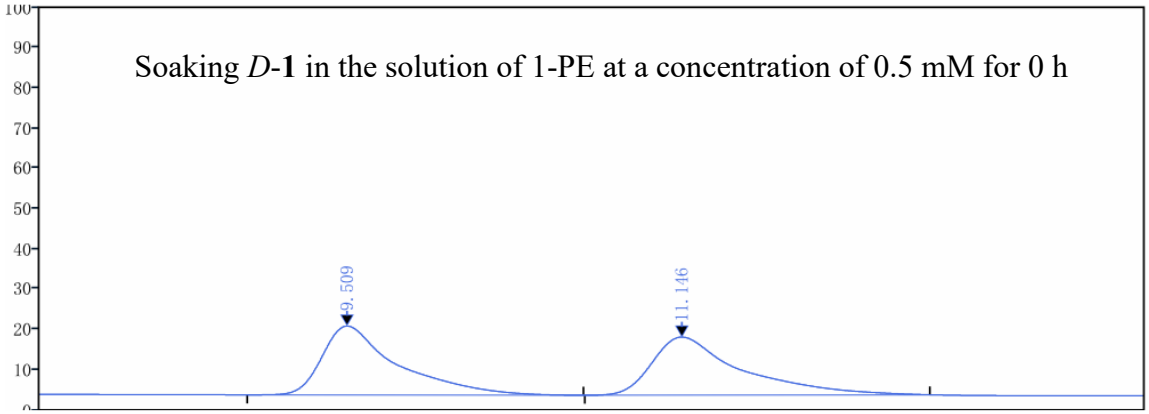




Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =96/4; flow rate =1.0 mL/min; 220 nm.



Serial Number	Type	Retention Time [min]	Peak Area	Area %
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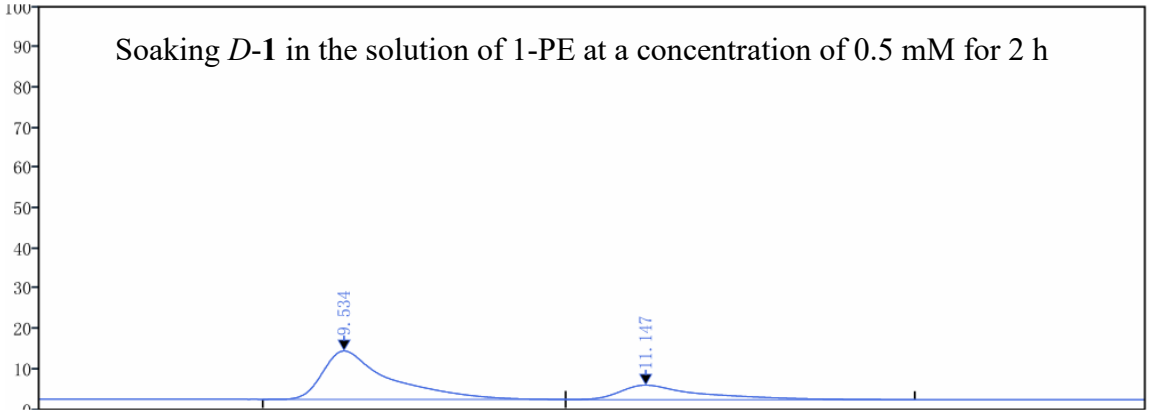
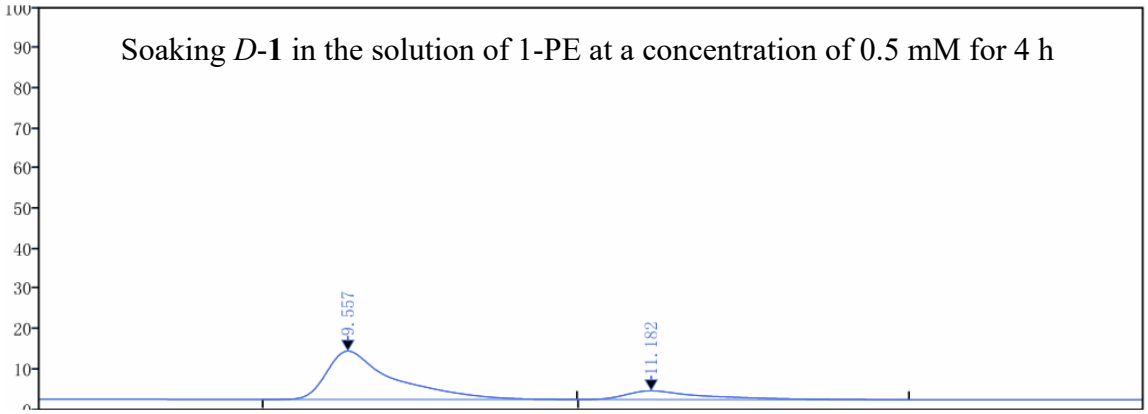
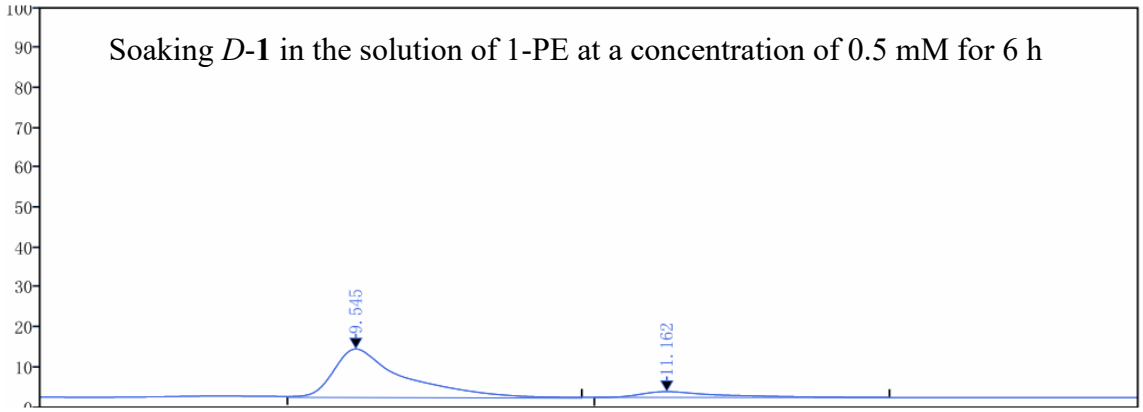
2	BM	11.146	782.38	49.78
The Total			1571.57	



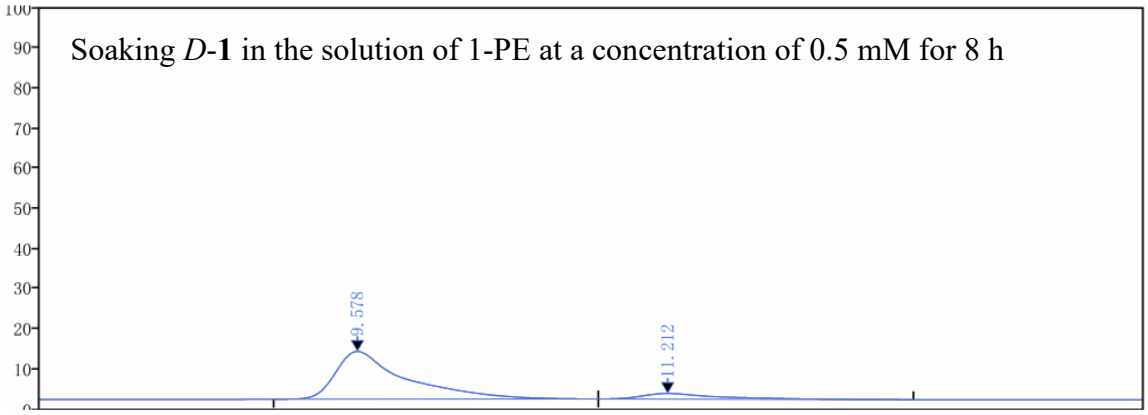
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BM	9.534	524.03	73.78
2	MB	11.147	186.22	26.22
The Total			710.25	



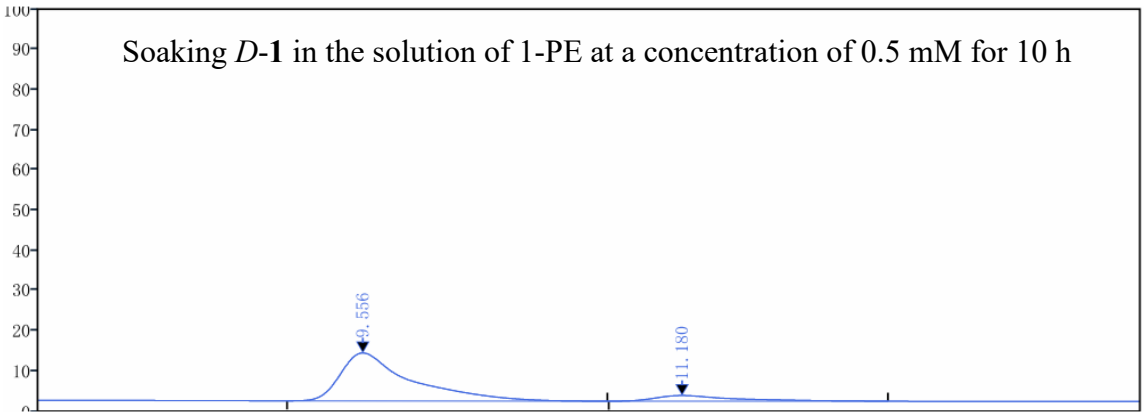
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BB	9.557	441.85	82.83
2	BB	11.182	91.61	17.17
The Total			533.46	



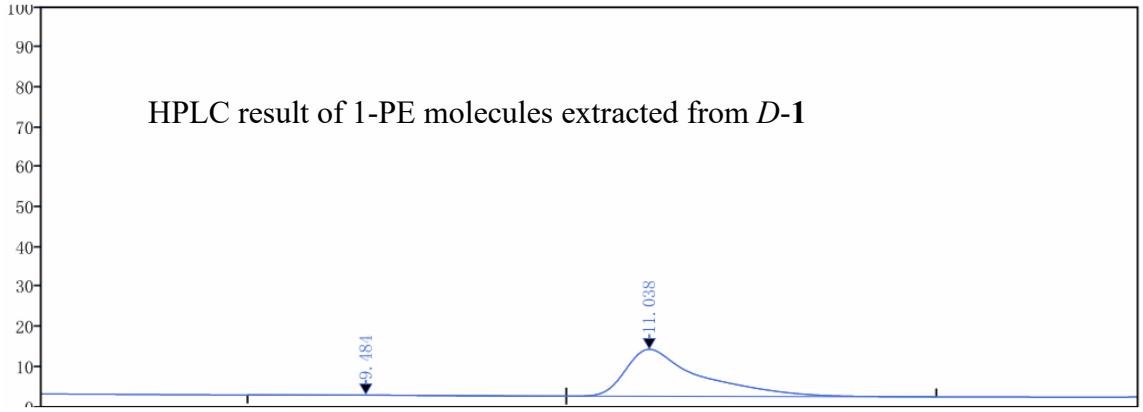
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.545	398.75	88.18
2	MM	11.162	53.45	11.82
The Total			452.20	



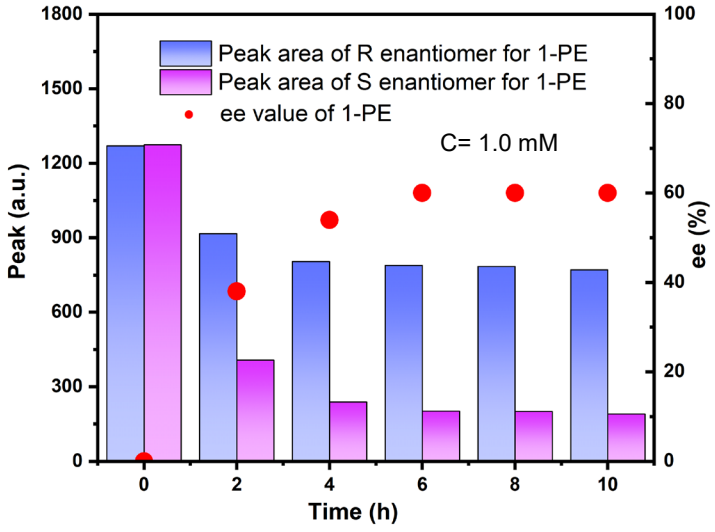
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.578	392.28	88.02
2	BM	11.212	53.40	11.98
The Total			445.68	

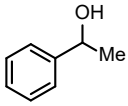


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BB	9.556	391.80	88.40
2	BM	11.180	51.43	11.60
The Total			443.23	

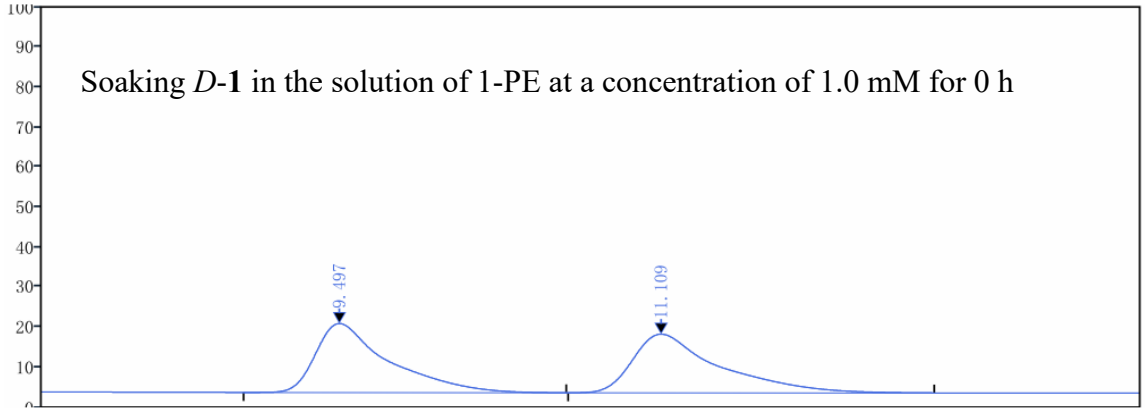


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.484	0.52	0.14
2	BM	11.038	380.95	99.86
The Total			381.47	

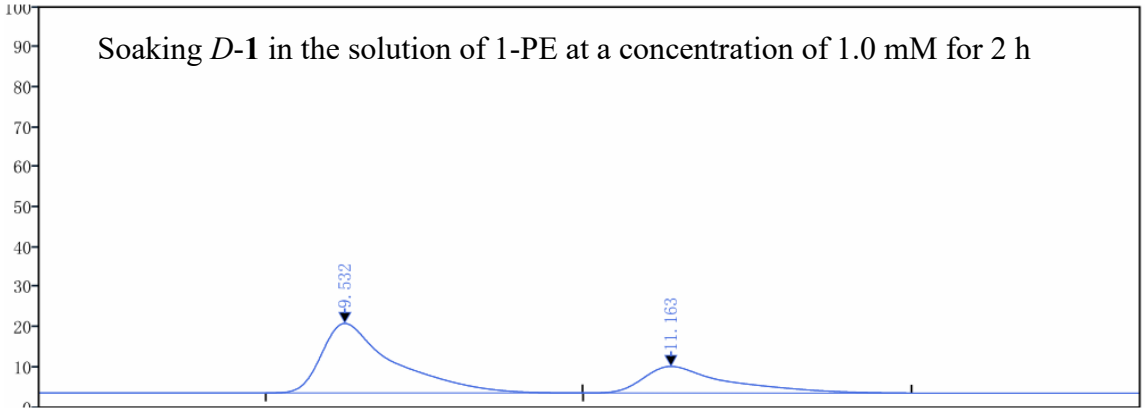




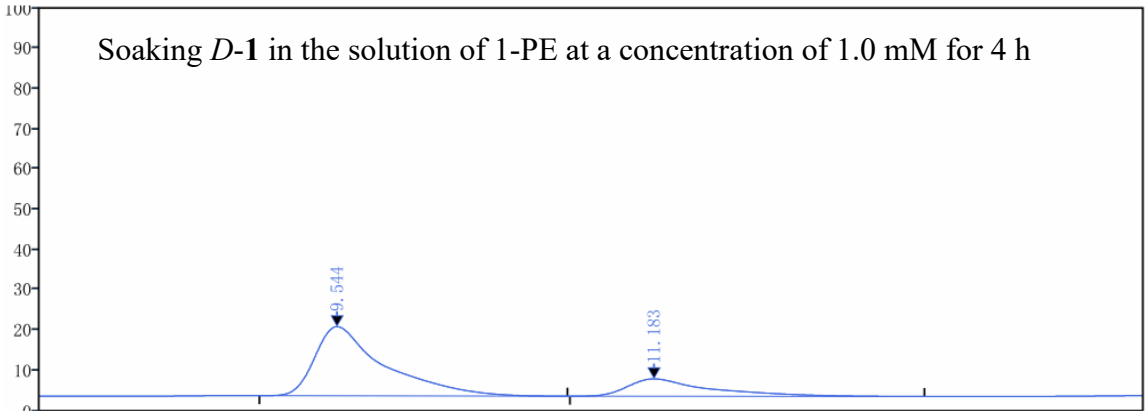
Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =96/4; flow rate =1.0 mL/min; 220 nm.



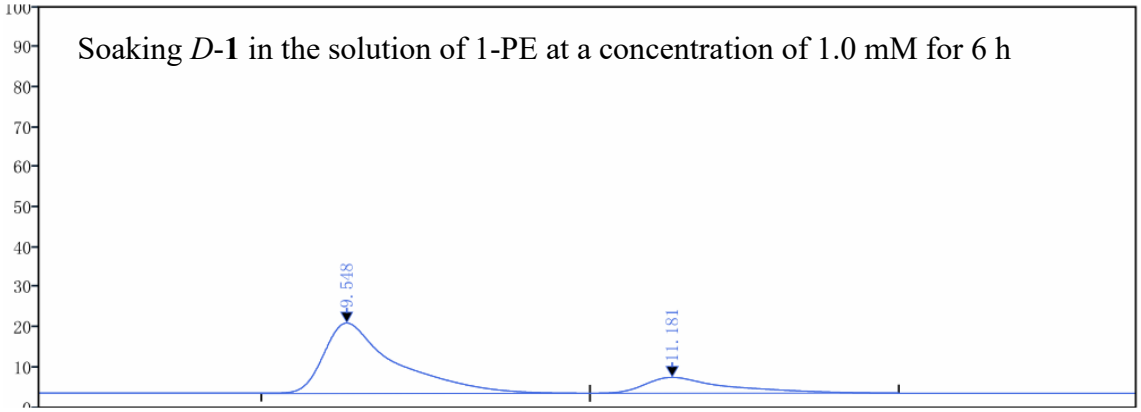
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BB	9.497	1270.03	49.92
2	BM	11.109	1274.17	50.08
The Total			2544.20	



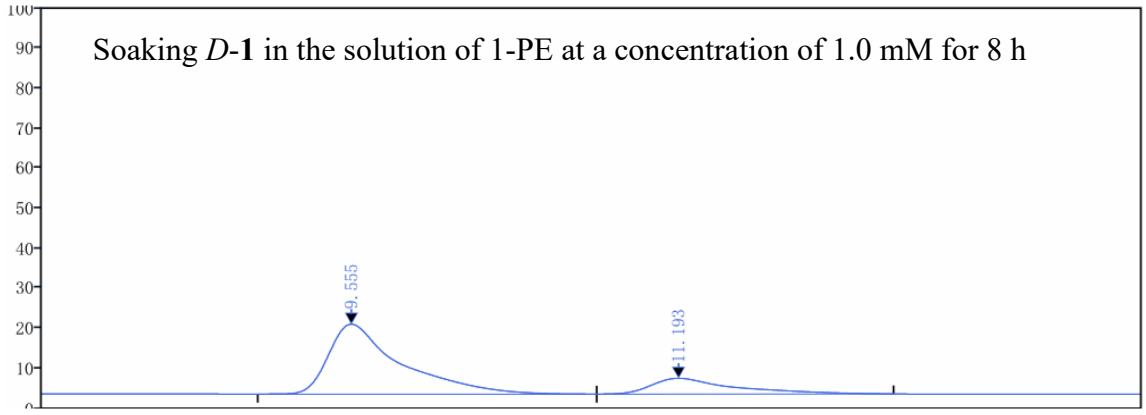
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MB	9.532	917.98	69.23
2	BM	11.163	407.98	30.77
The Total			1325.96	



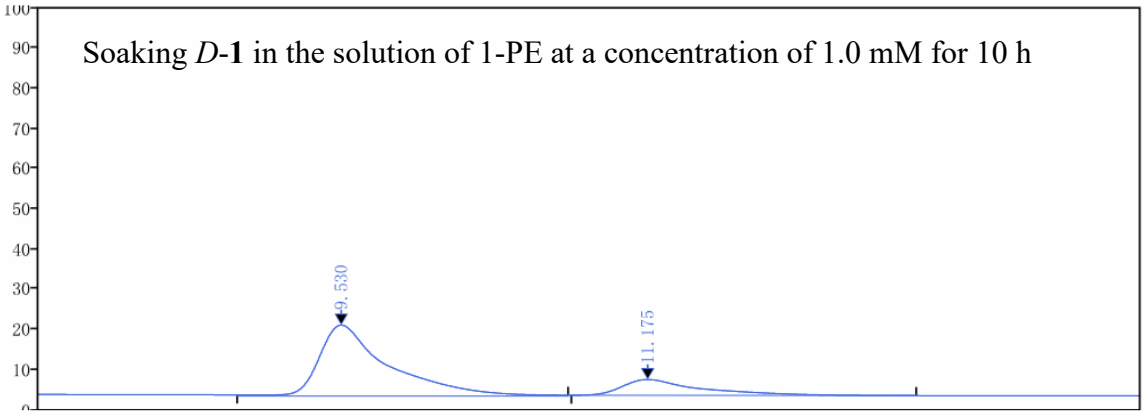
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BB	9.544	805.48	77.08
2	BB	11.183	239.57	22.92
The Total			1045.05	



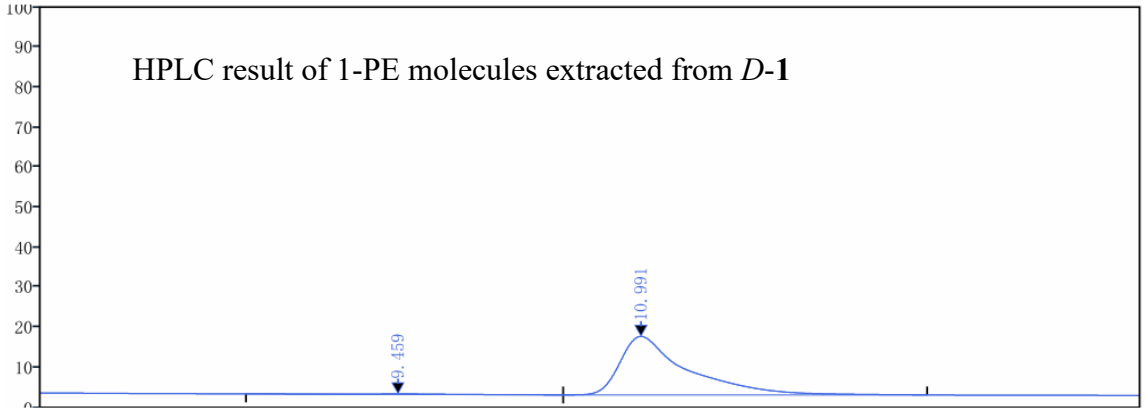
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.548	789.67	79.61
2	BM	11.181	202.27	20.39
The Total			991.94	



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MB	9.555	785.66	79.59
2	BM	11.193	201.51	20.41
The Total			987.17	

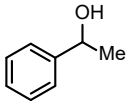


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.530	771.40	80.15
2	MM	11.175	190.99	19.85
The Total			962.39	

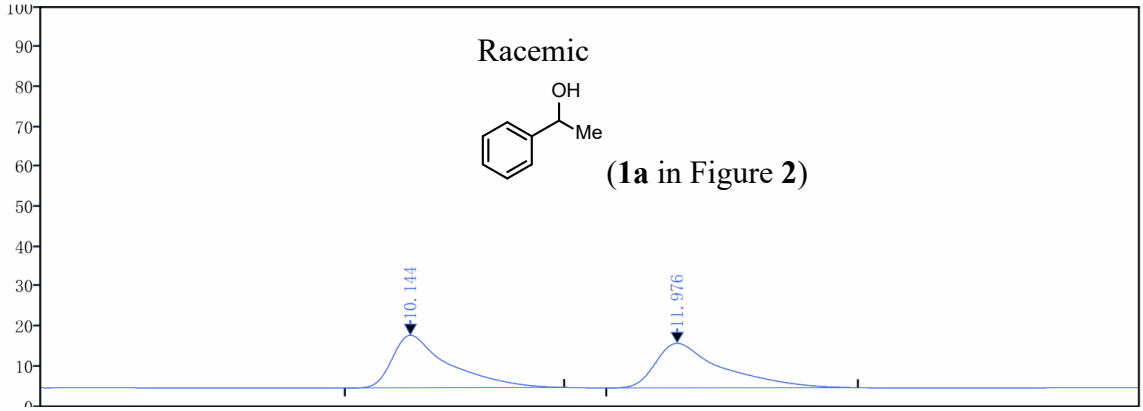


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	9.459	1.73	0.31
2	BM	10.991	558.10	99.69
The Total			559.83	

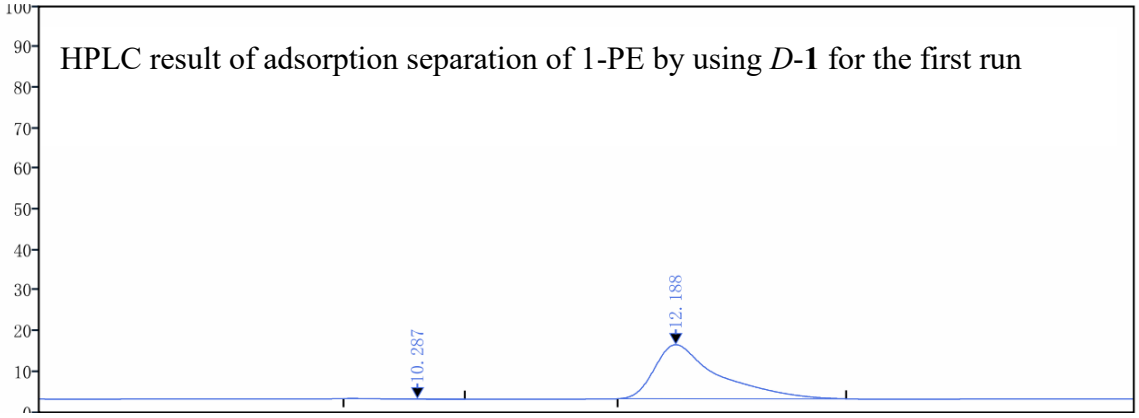
13. Figure S8. HPLC results of chiral molecules resolved by *D*-1 (in Figure 2 and 3).



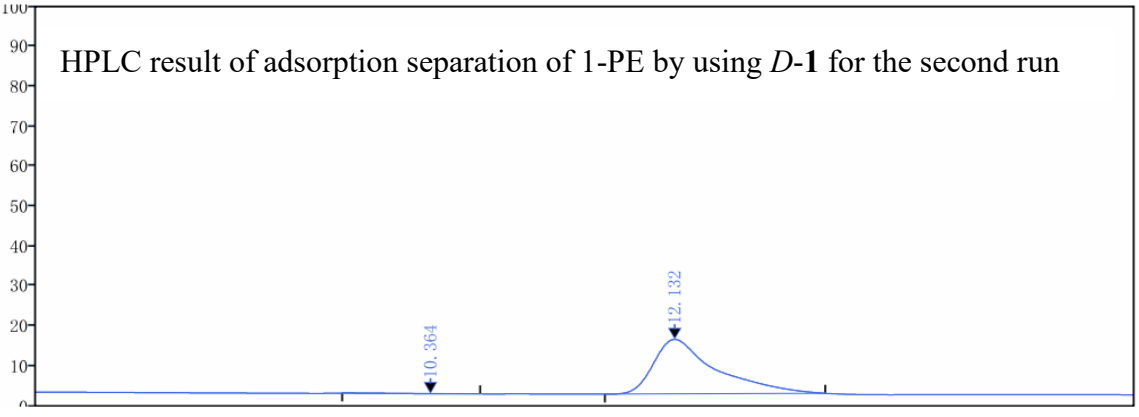
Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =96/4; flow rate =1.0 mL/min; 220 nm;



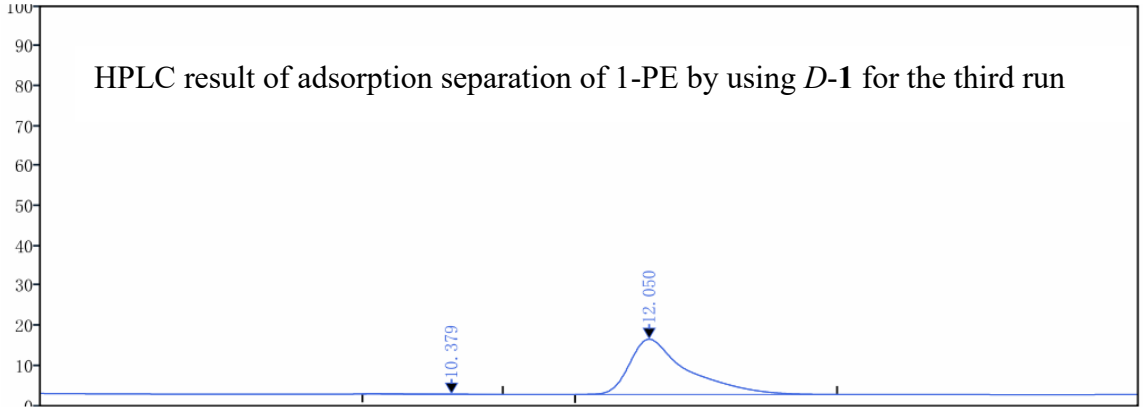
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	10.144	675.95	49.99
2	BM	11.976	676.16	50.01
The Total			1352.11	



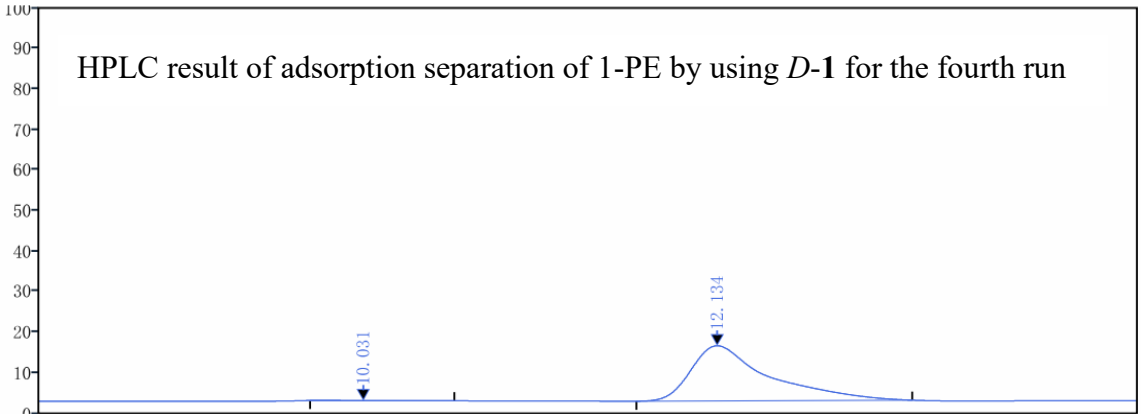
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	10.287	0.60	0.09
2	MM	12.188	691.87	99.91
The Total			692.47	



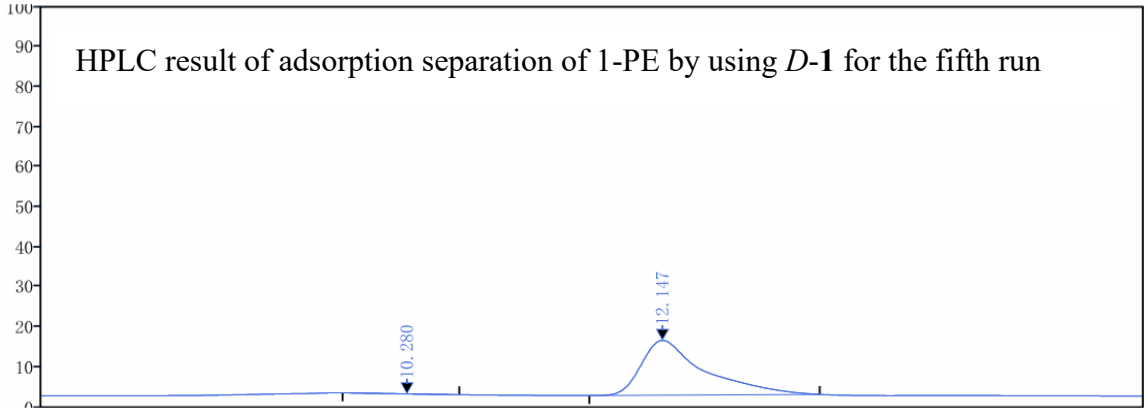
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	10.364	0.80	0.21
2	BM	12.132	388.26	99.79
The Total			389.06	



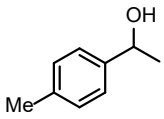
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	10.379	2.21	0.18
2	BB	12.050	1252.99	99.82
The Total			1255.20	



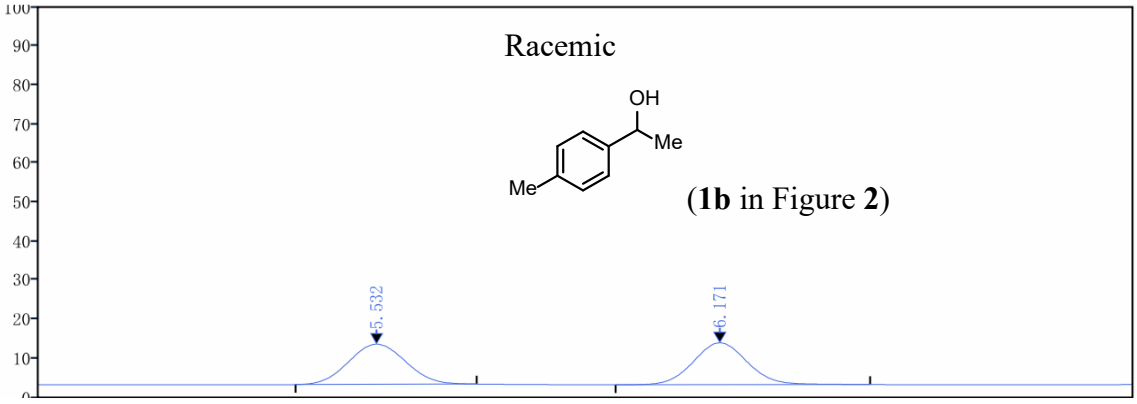
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	10.031	0.44	0.06
2	MM	12.134	767.23	99.94
The Total			767.67	



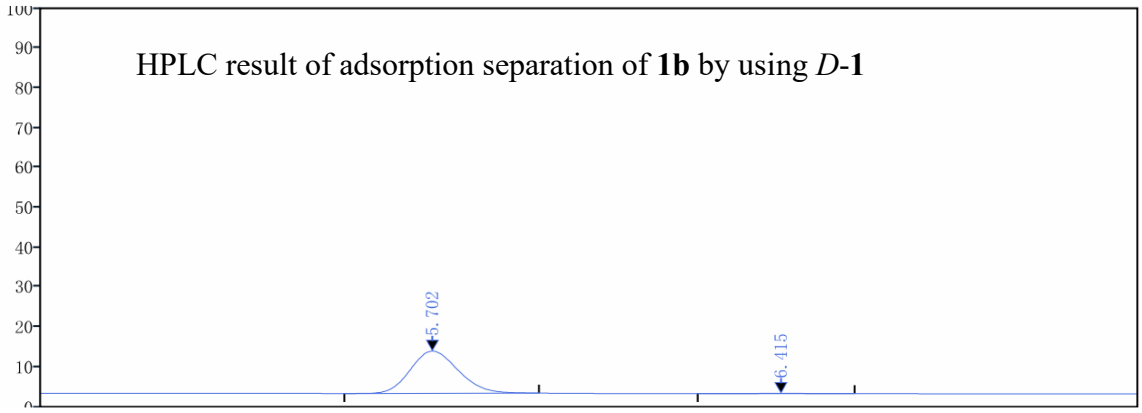
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	10.280	0.78	0.12
2	BM	12.147	675.73	99.88
The Total			676.51	



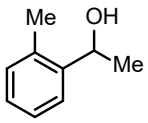
Chiral HPLC analysis: Daicel Chiralce AD: hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 220 nm;



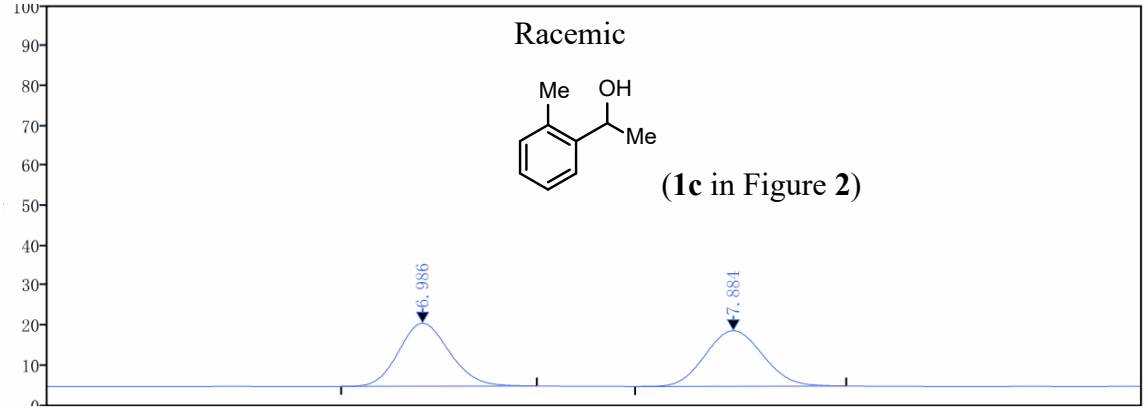
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.532	1793.80	50.30
2	BM	6.171	1772.62	49.70
The Total			3566.42	



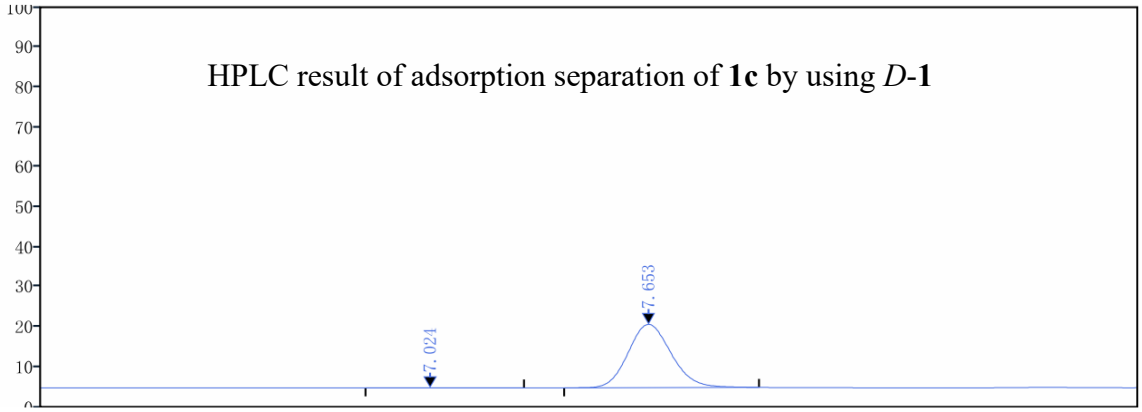
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.702	1045.70	99.15
2	MM	6.415	8.99	0.85
The Total			1054.69	



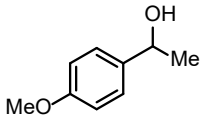
Chiral HPLC analysis: Daicel Chiralce AD: hexane/*i*-PrOH =97/3; flow rate =1.0 mL/min; 220 nm;



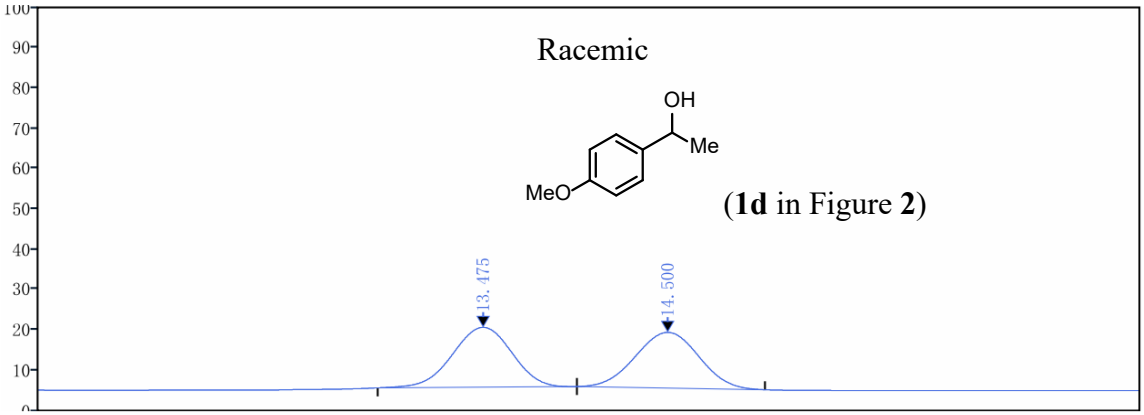
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	6.986	2758.14	50.07
2	BM	7.884	2749.99	49.93
The Total			5508.13	



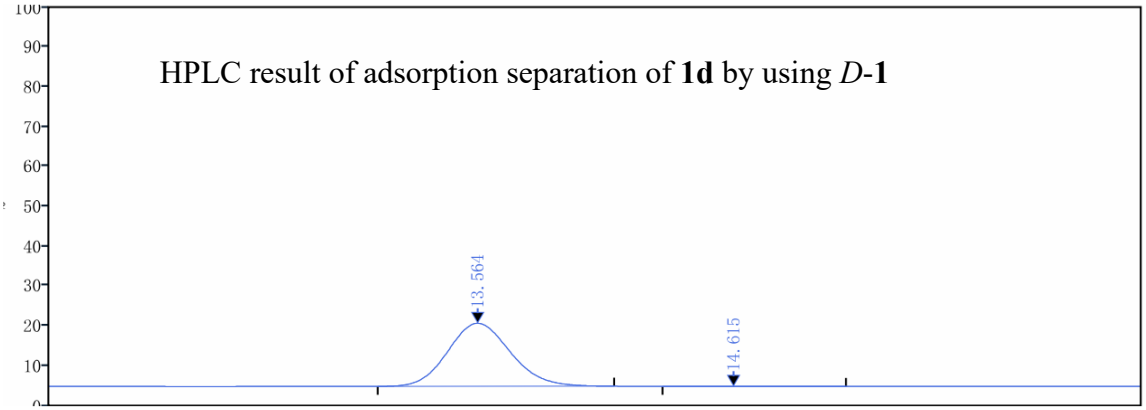
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	7.024	0.80	0.02
2	BM	7.653	3240.77	99.98
The Total			3241.57	



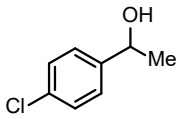
Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =97/3; flow rate =1.0 mL/min; 220 nm;



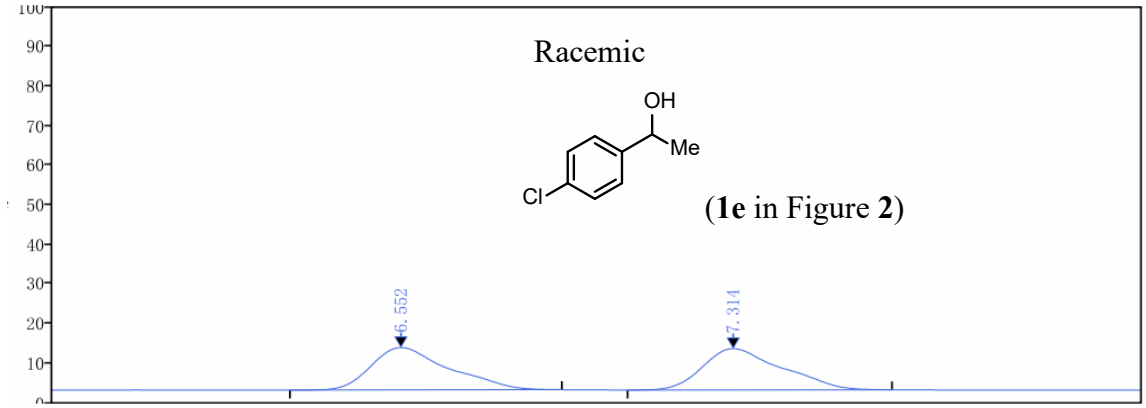
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	13.475	2018.40	50.05
2	MM	14.500	2014.61	49.95
The Total			4033.01	



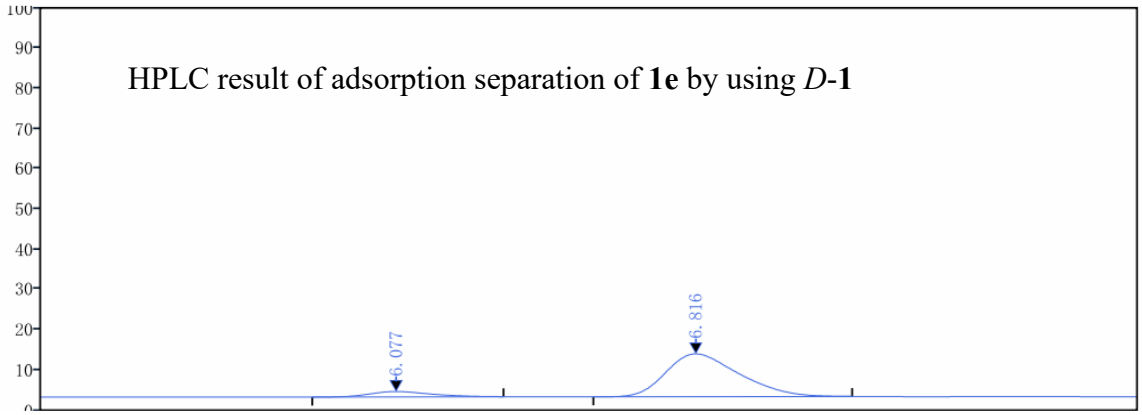
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	13.564	603.80	99.89
2	MM	14.615	0.63	0.11
The Total			604.43	



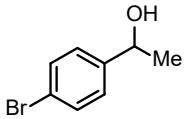
Chiral HPLC analysis: Daicel Chiralce AS hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 230nm;



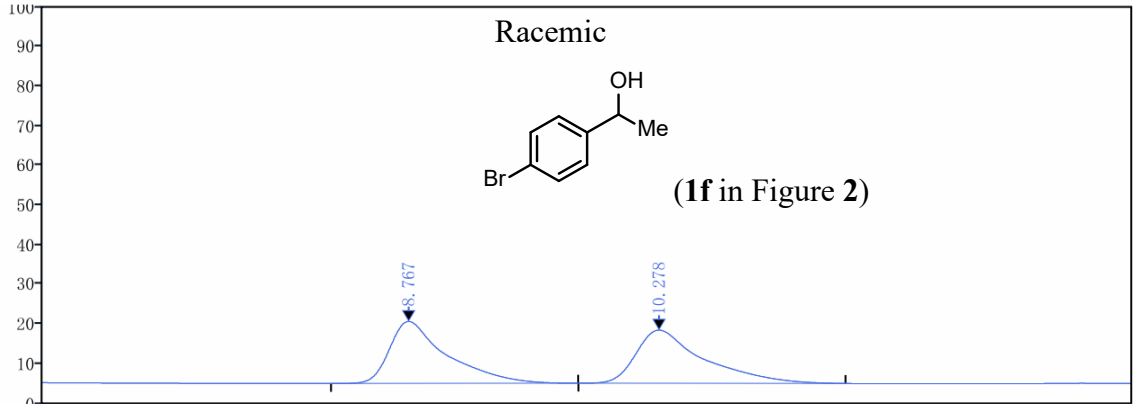
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BM	6.552	2817.16	50.24
2	BM	7.314	2789.74	49.76
The Total			5606.90	



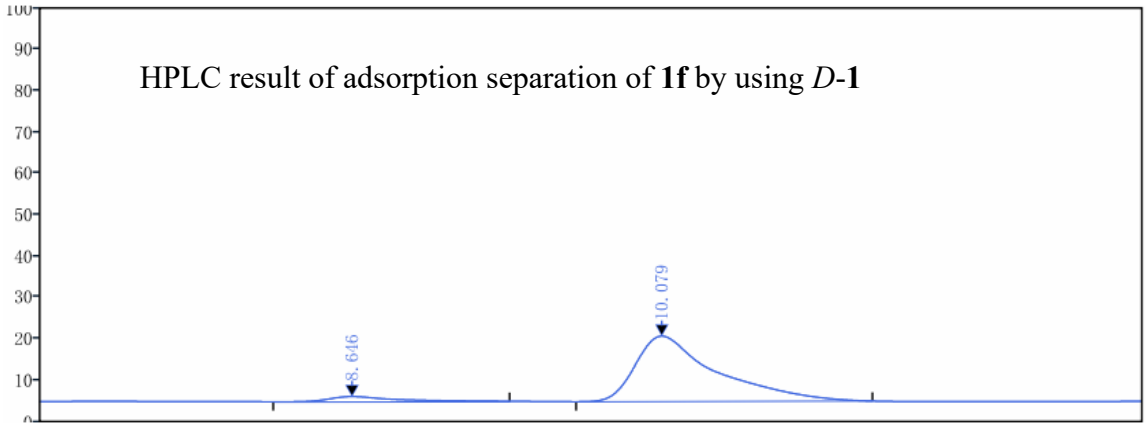
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BM	6.077	92.67	9.47
2	MM	6.816	885.87	90.53
The Total			978.54	



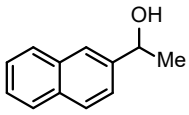
Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 220 nm;



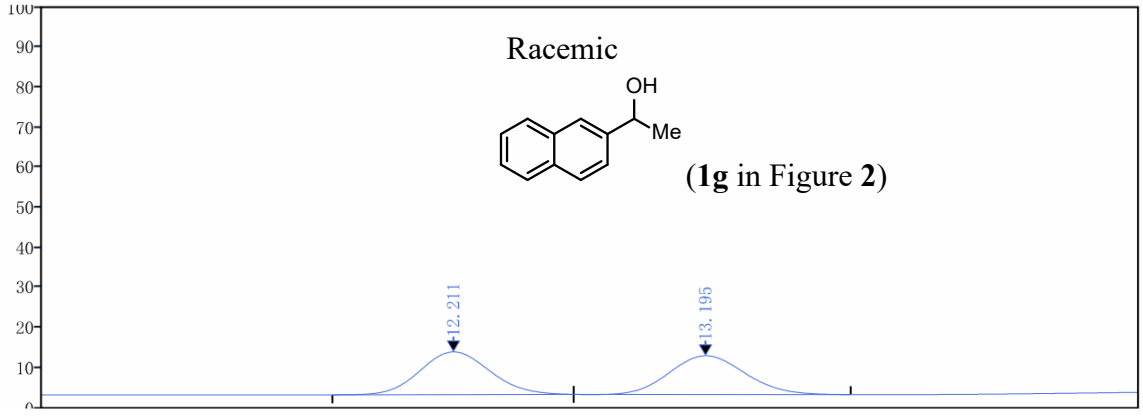
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BB	8.767	1723.27	49.60
2	BM	10.278	1751.20	50.40
The Total			3474.47	



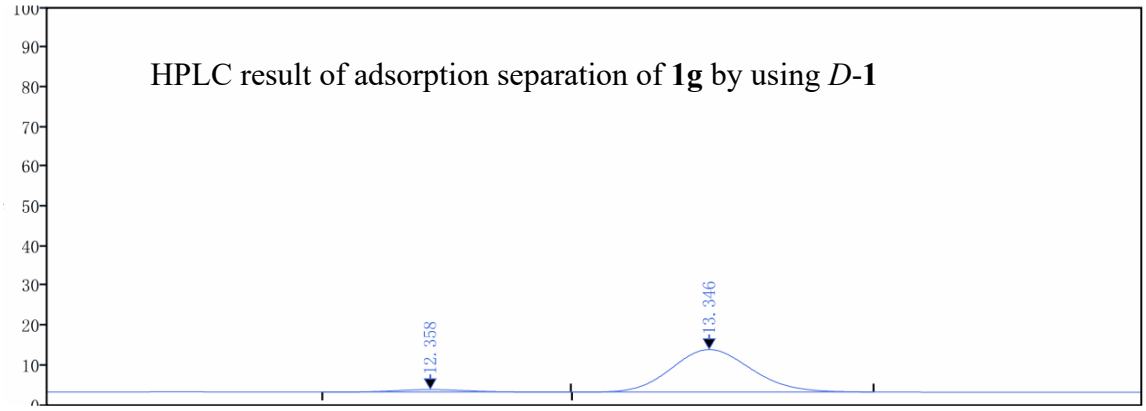
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.646	110.30	7.40
2	BM	10.079	1380.77	92.60
The Total			1491.07	



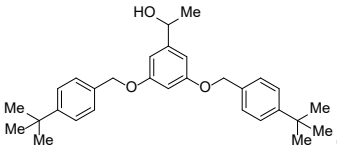
Chiral HPLC analysis: Daicel Chiralce OJ: hexane/*i*-PrOH =90/10; flow rate =1.0 mL/min; 220 nm;



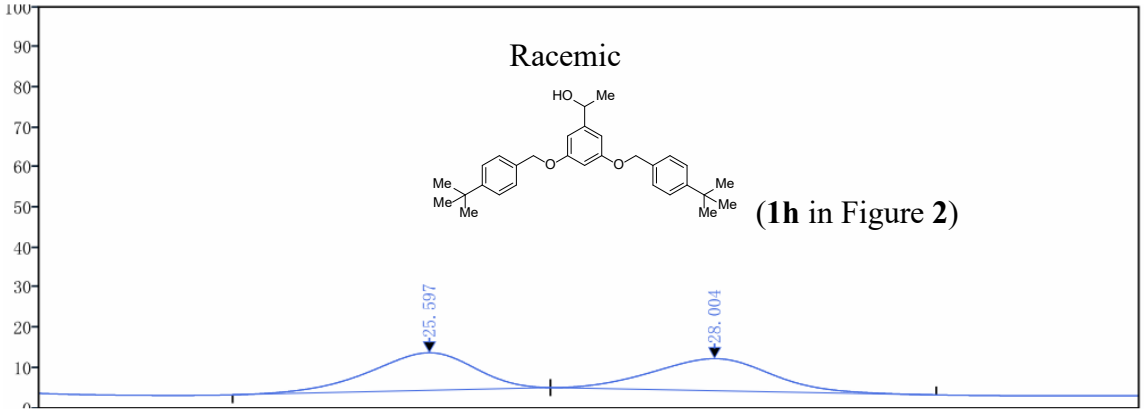
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	12.211	1074.15	50.48
2	MM	13.195	1053.64	49.52
The Total			2127.79	



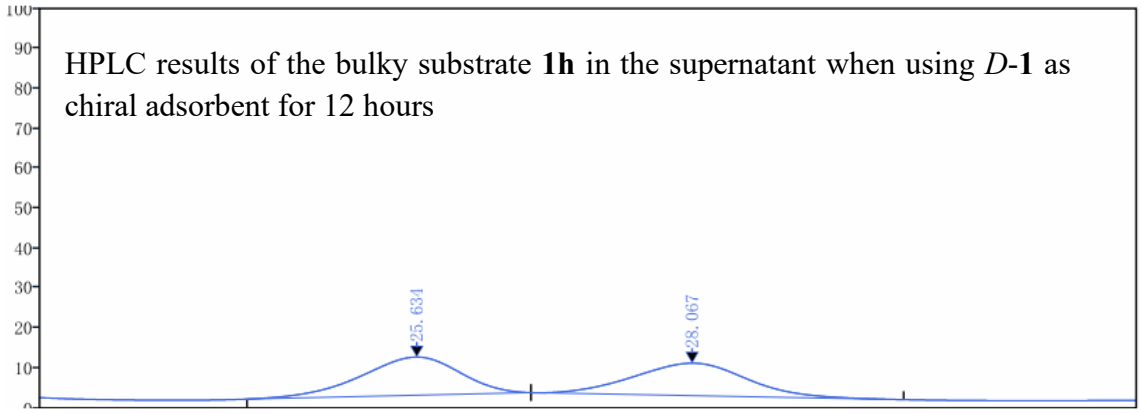
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BB	12.358	56.09	5.15
2	BM	13.346	1034.07	94.85
The Total			1090.16	



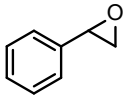
Chiral HPLC analysis: Daicel Chiralce IA: hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 220 nm.



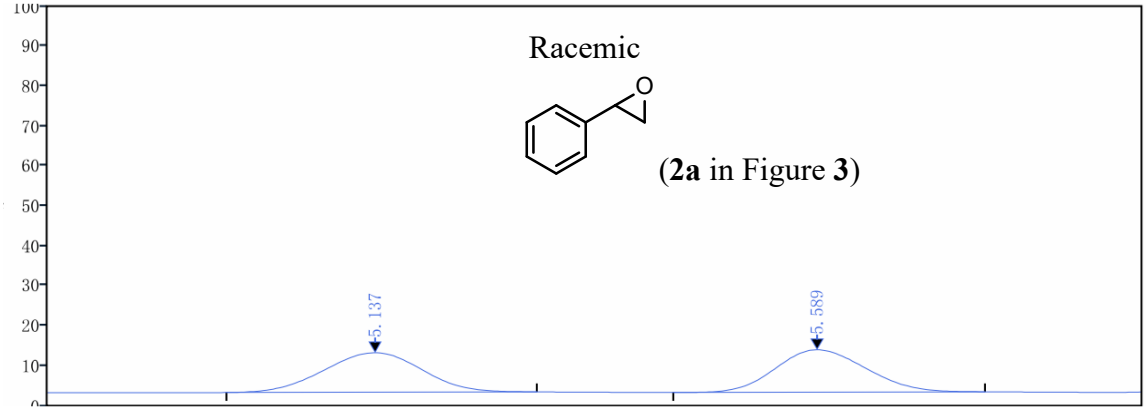
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	25.597	2992.83	50.90
2	MM	28.004	2886.63	49.10
The Total			5879.46	



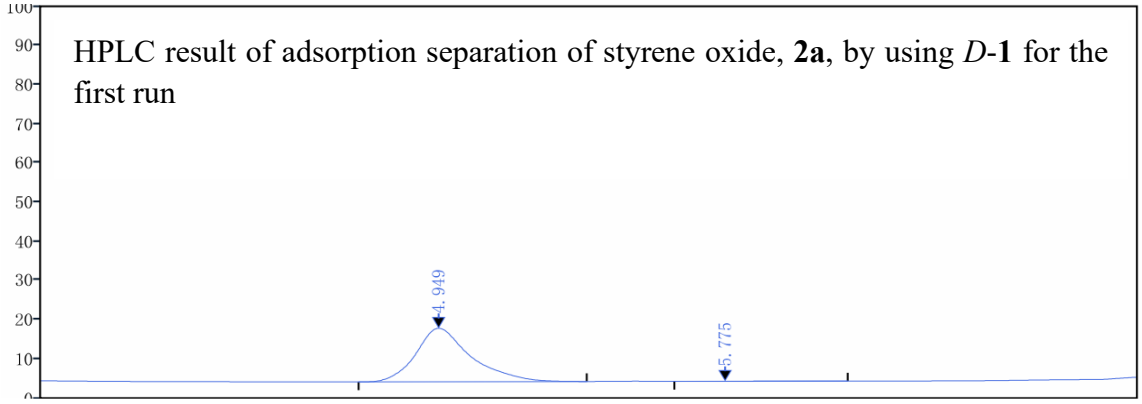
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	25.634	2879.63	50.55
2	MM	28.067	2816.98	49.45
The Total			5696.61	



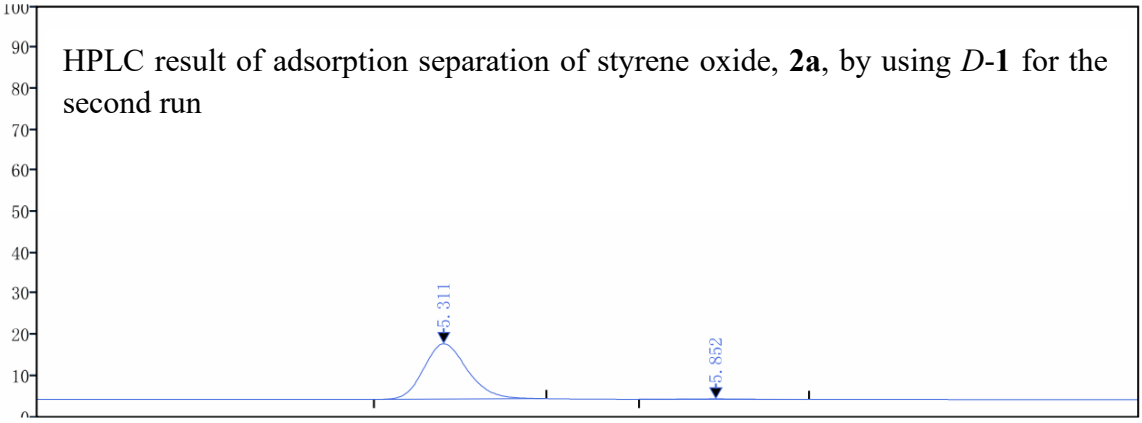
Chiral HPLC analysis: Daicel Chiralce IC: hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 220 nm;



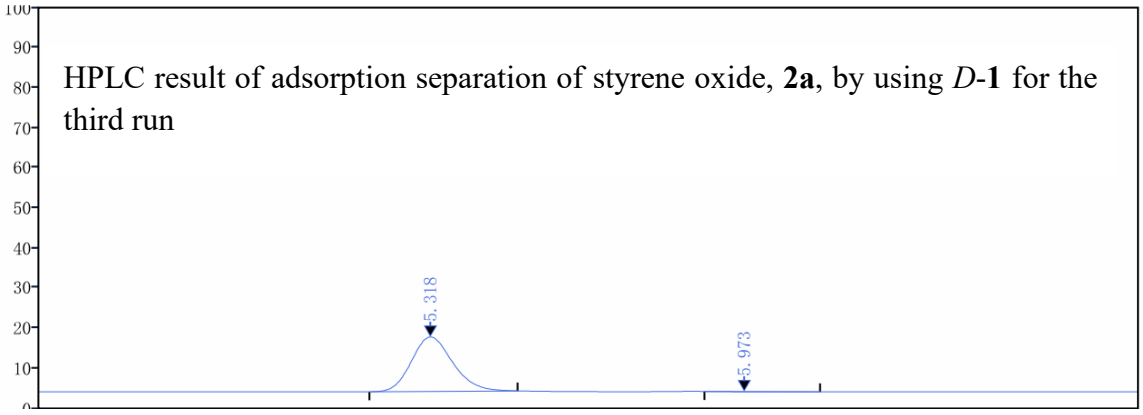
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.137	2394.39	50.29
2	BM	5.589	2366.98	49.71
The Total			4761.37	



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BB	4.949	1053.24	99.92
2	MM	5.775	0.87	0.08
The Total			1054.11	

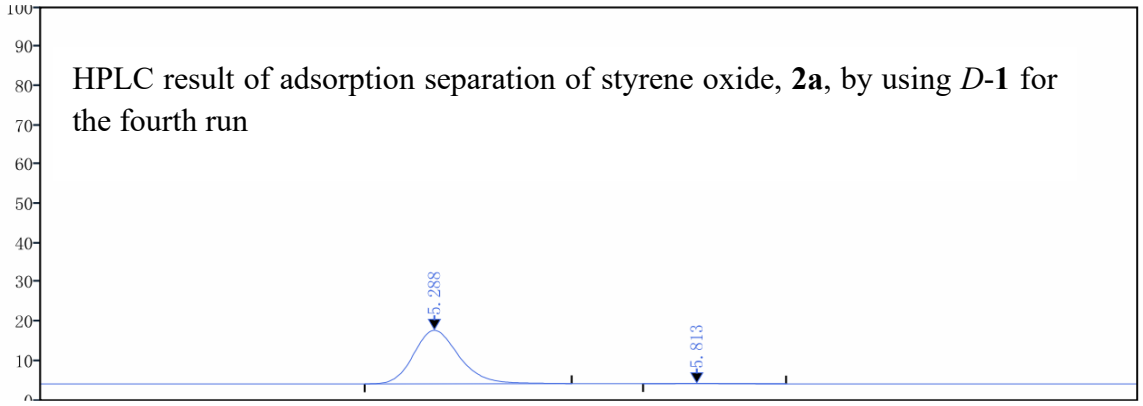


Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.311	625.97	99.69
2	MM	5.852	1.96	0.31
The Total			627.93	



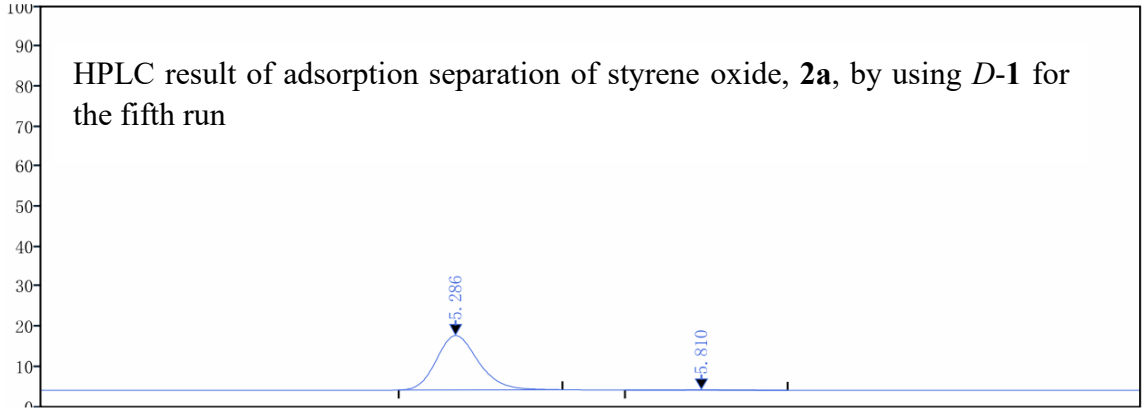
HPLC result of adsorption separation of styrene oxide, **2a**, by using *D*-**1** for the third run

Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.318	619.54	99.54
2	MM	5.973	2.88	0.46
The Total			622.42	

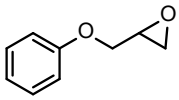


HPLC result of adsorption separation of styrene oxide, **2a**, by using *D*-**1** for the fourth run

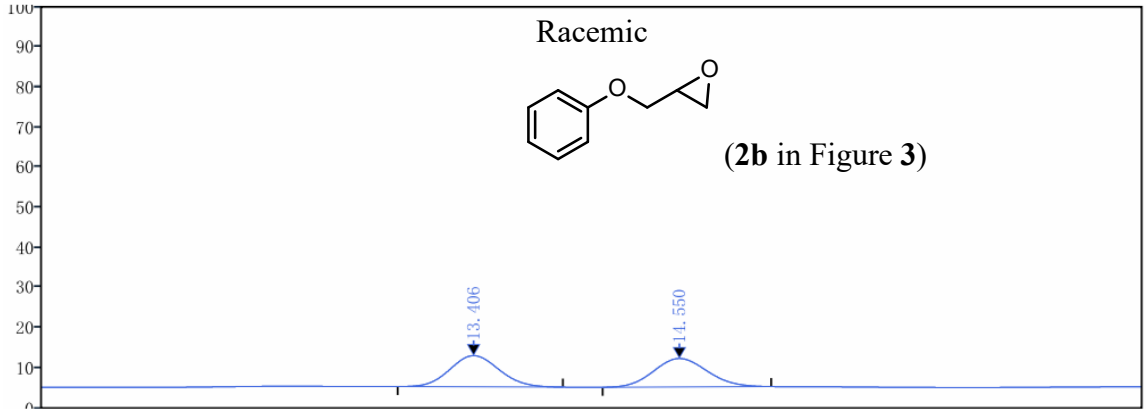
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.288	962.89	99.86
2	MM	5.813	1.33	0.14
The Total			964.22	



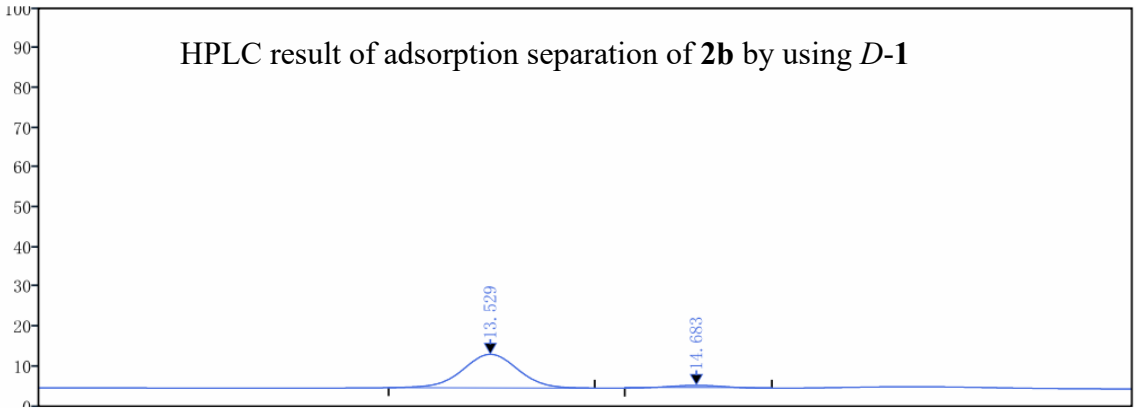
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	5.286	834.92	99.52
2	MM	5.810	4.05	0.48
The Total			838.97	



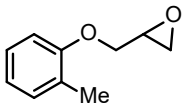
Chiral HPLC analysis: Daicel Chiralce AS: hexane/*i*-PrOH =98/2; flow rate =1.0 mL/min; 220 nm;



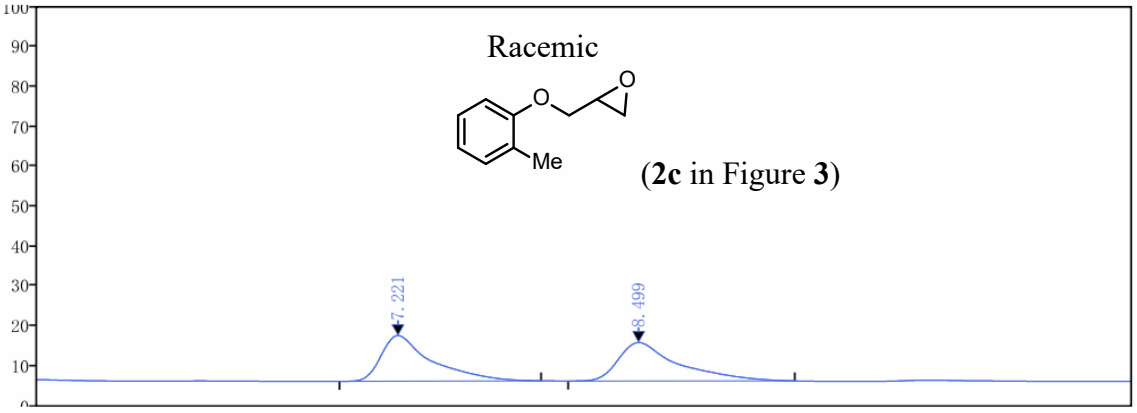
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	13.406	811.88	50.15
2	MM	14.550	807.14	49.85
The Total			1619.02	



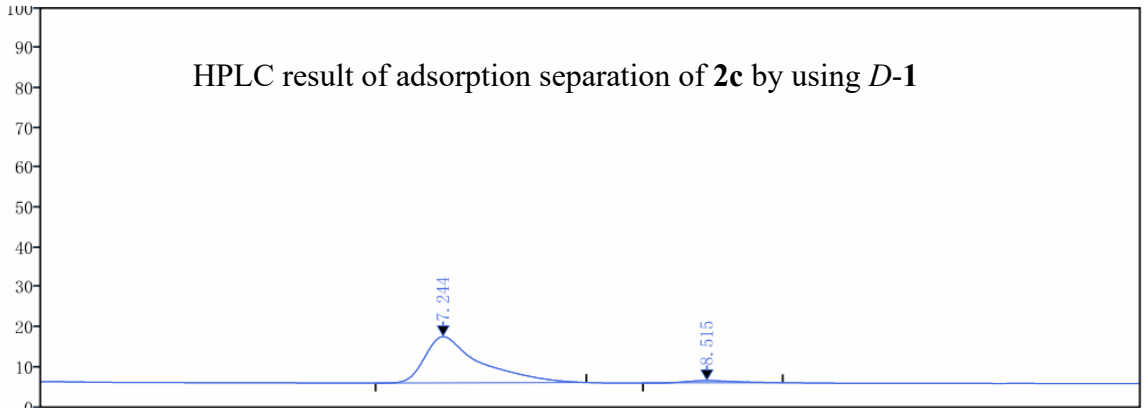
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	13.529	508.95	96.25
2	MM	14.683	19.81	3.75
The Total			528.76	



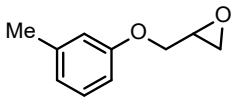
Chiral HPLC analysis: Daicel Chiralce IA: hexane/*i*-PrOH =98/2; flow rate =1.0 mL/min; 220 nm;



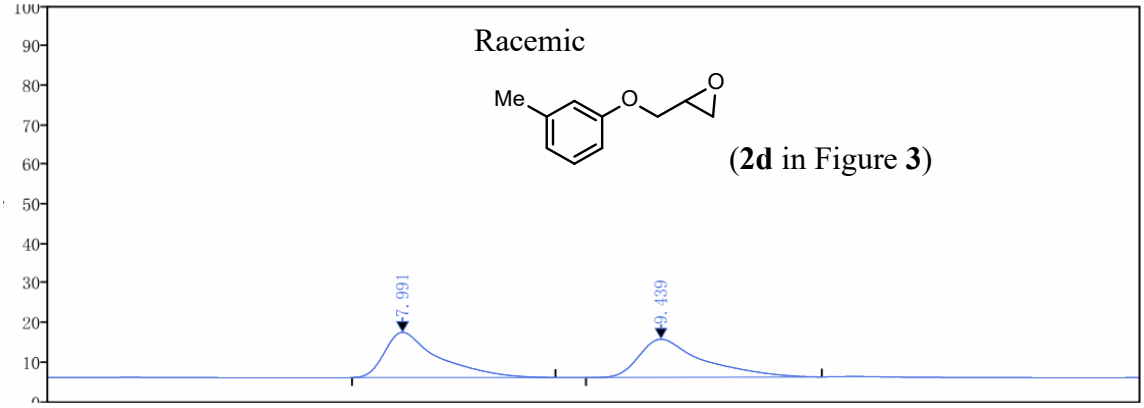
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BM	7.221	771.51	50.10
2	BM	8.499	768.53	49.90
The Total			1540.04	



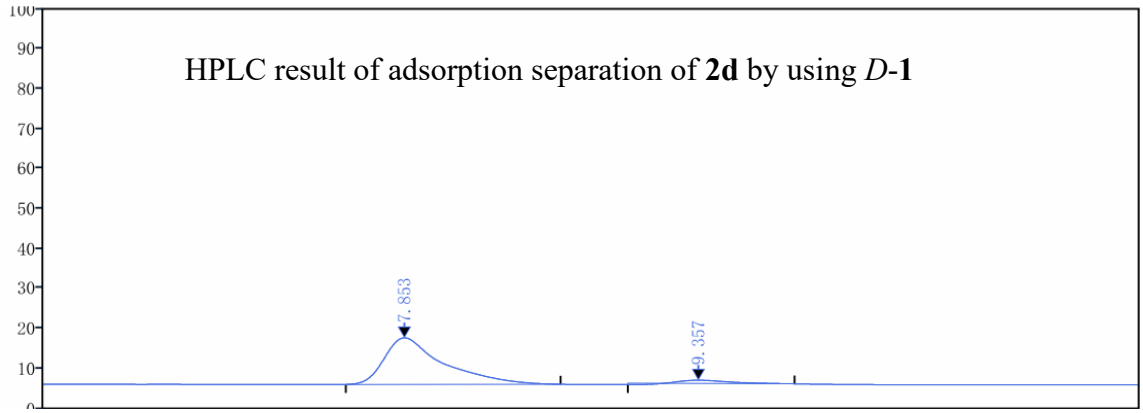
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BM	7.244	334.21	96.42
2	MM	8.515	12.39	3.58
The Total			346.60	



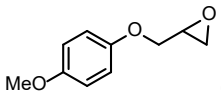
Chiral HPLC analysis: Daicel Chiralce AS: hexane/*i*-PrOH =98/2; flow rate =1.0 mL/min; 220 nm;



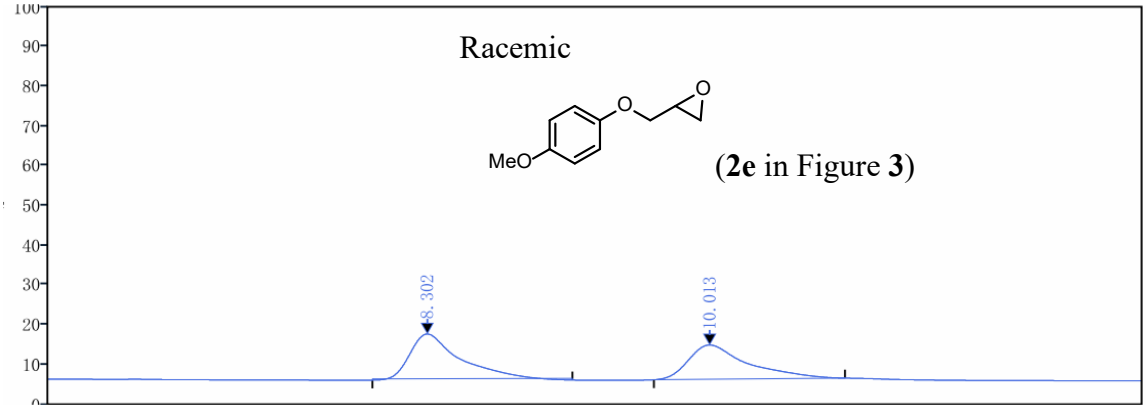
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	7.991	816.77	50.07
2	BB	9.439	814.60	49.93
The Total			1631.37	



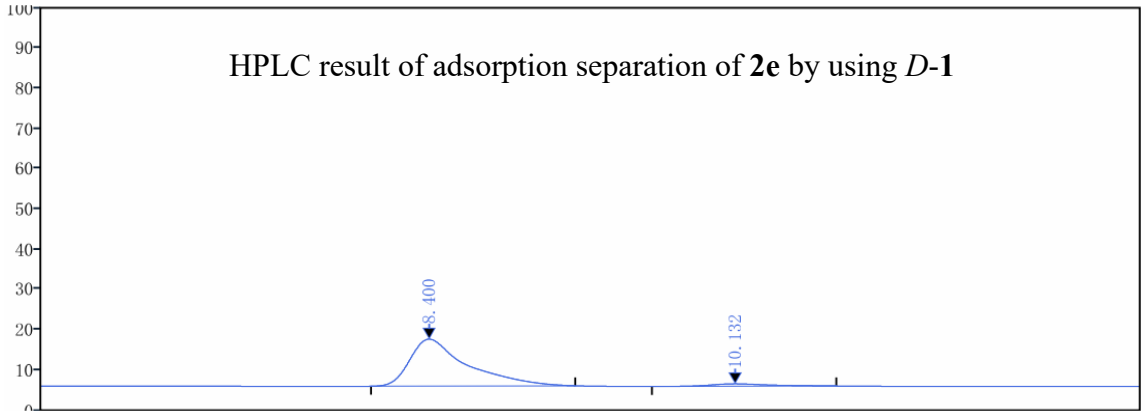
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	7.853	1006.97	96.15
2	MM	9.357	40.33	3.85
The Total			1047.30	



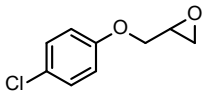
Chiral HPLC analysis: Daicel Chiralce OD: hexane/*i*-PrOH =90/10; flow rate =1.0 mL/min; 254 nm;



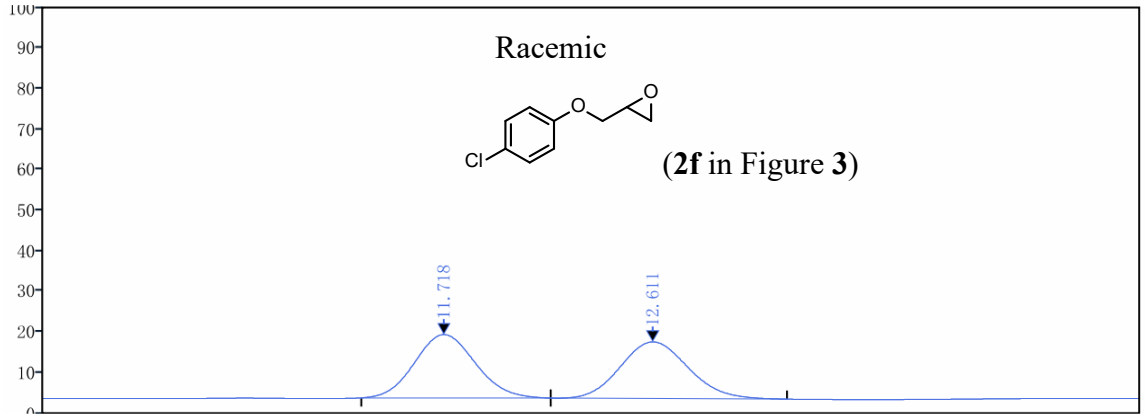
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.302	139.79	51.56
2	MM	10.013	131.36	48.44
The Total			271.15	



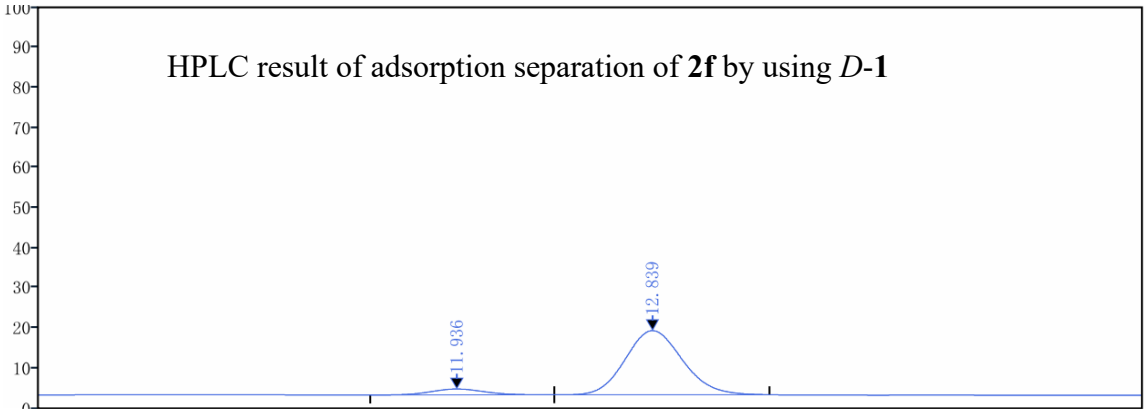
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	8.400	1442.12	95.17
2	BM	10.132	73.21	4.83
The Total			1515.33	



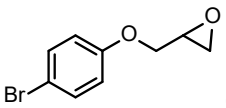
Chiral HPLC analysis: Daicel Chiralce OJ: hexane/*i*-PrOH =95/5; flow rate =1.0 mL/min; 220 nm;



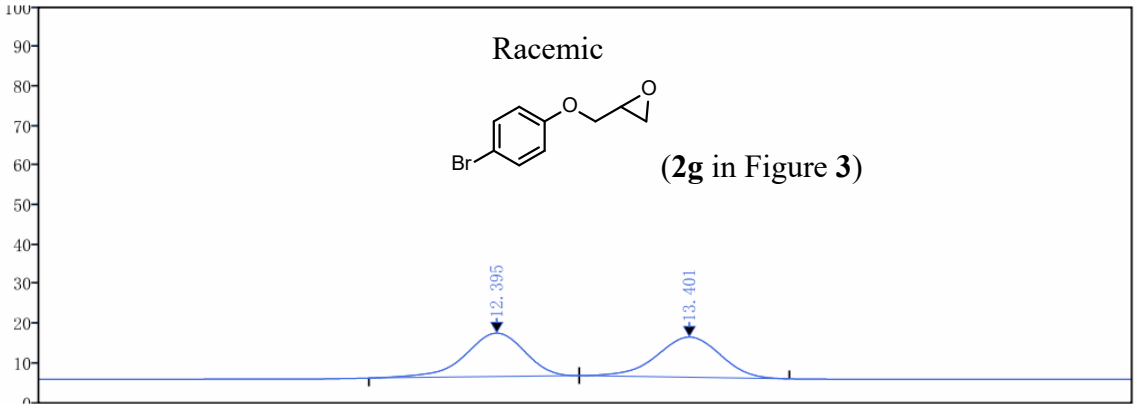
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	11.718	818.13	50.08
2	MM	12.611	815.59	49.92
The Total			1633.72	



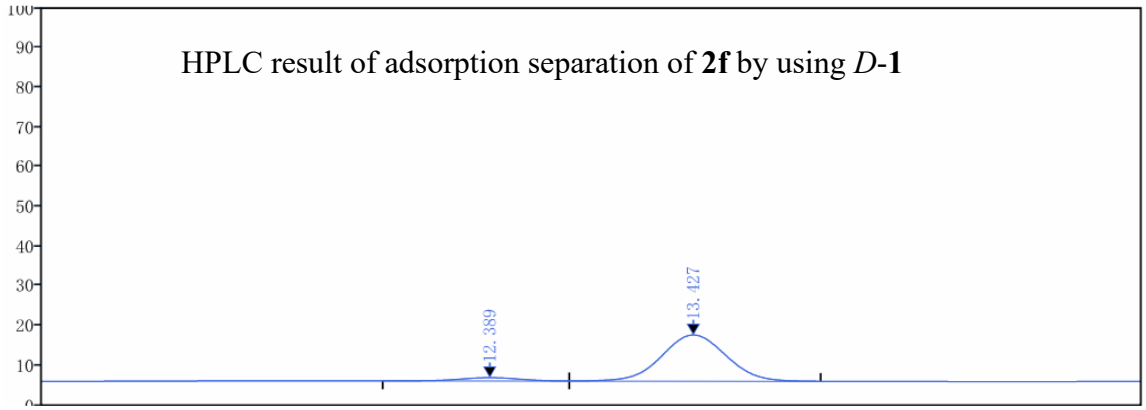
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BB	11.936	73.00	7.84
2	BM	12.839	858.05	92.16
The Total			931.05	



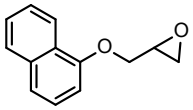
Chiral HPLC analysis: Daicel Chiralce OJ: hexane/*i*-PrOH =90/10; flow rate =1.0 mL/min; 220 nm;



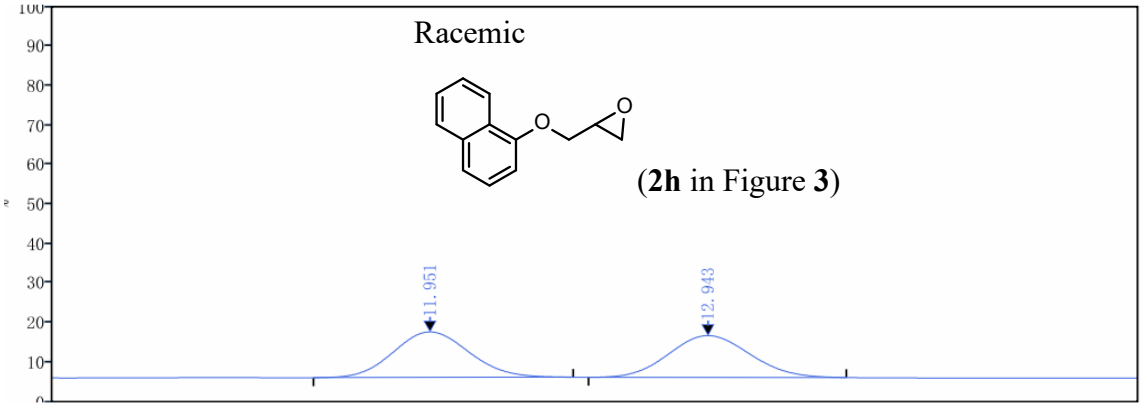
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	12.395	1896.34	50.16
2	MM	13.401	1883.87	49.84
The Total			3780.21	



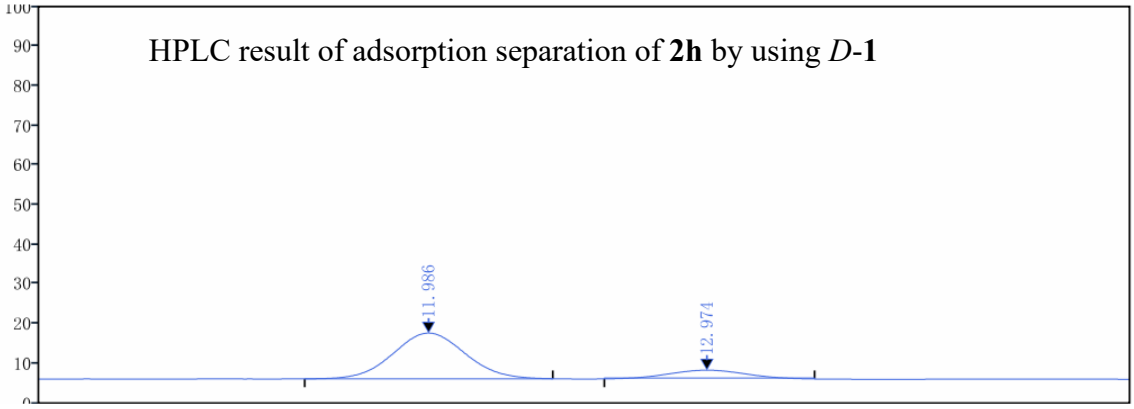
Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	BM	12.389	118.11	6.11
2	MM	13.427	1814.32	93.89
The Total			1932.43	



Chiral HPLC analysis: Daicel Chiralce AS: hexane/*i*-PrOH =98/2; flow rate =1.0 mL/min; 220 nm;



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	11.951	1288.13	50.09
2	MM	12.943	1283.38	49.91
The Total			2571.51	



Serial Number	Type	Retention Time [min]	Peak Area	Area %
1	MM	11.986	272.50	86.54
2	MM	12.974	42.40	13.46
The Total			314.90	

14. Figure S9. Structural drawing of asymmetric unit of *D*-1.