

Eluent Tolerance and Enantioseparation Recovery of Chiral Packing Materials Based on Chitosan Bis(Phenylcarbamate)-(*n*-Octyl Urea)s for High Performance Liquid Chromatography

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1. ¹H-NMR Spectra Of Chitosan and Chitosan Derivatives

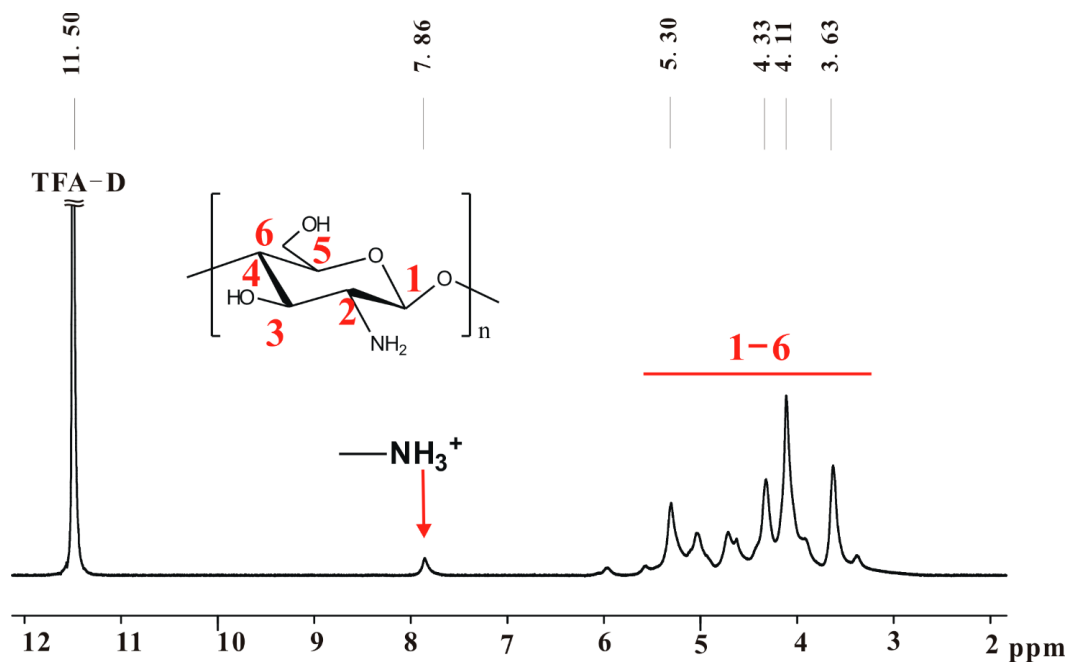


Figure S1. ¹H-NMR spectrum of chitosan (Ia) with the M_v of 1.5×10^5 .

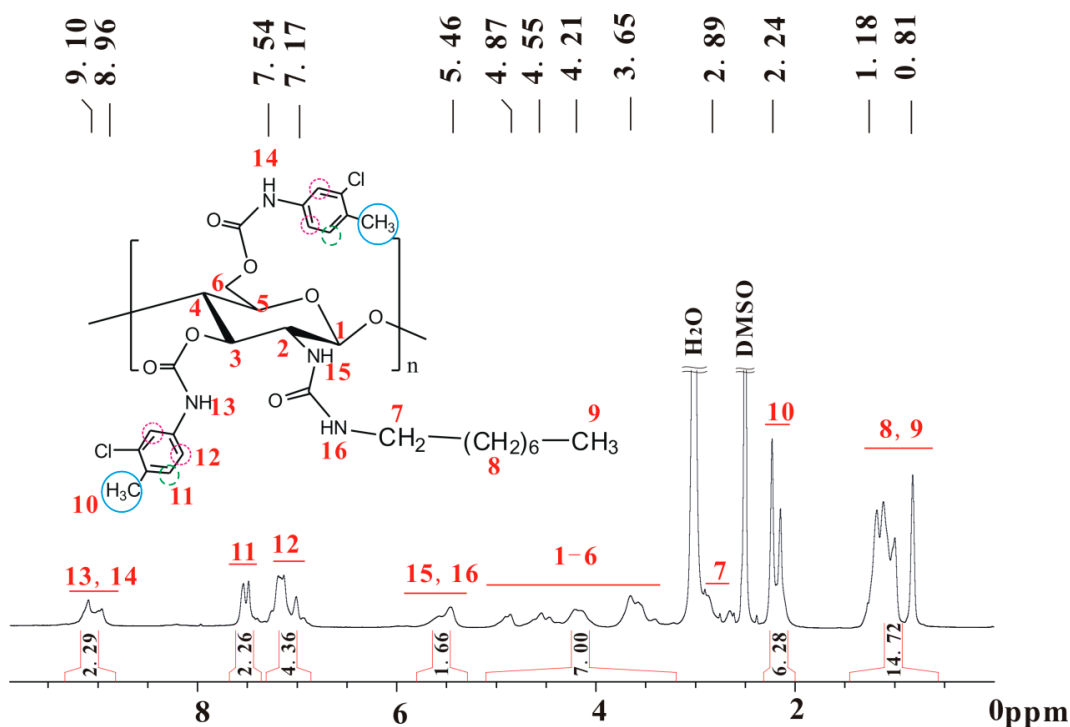
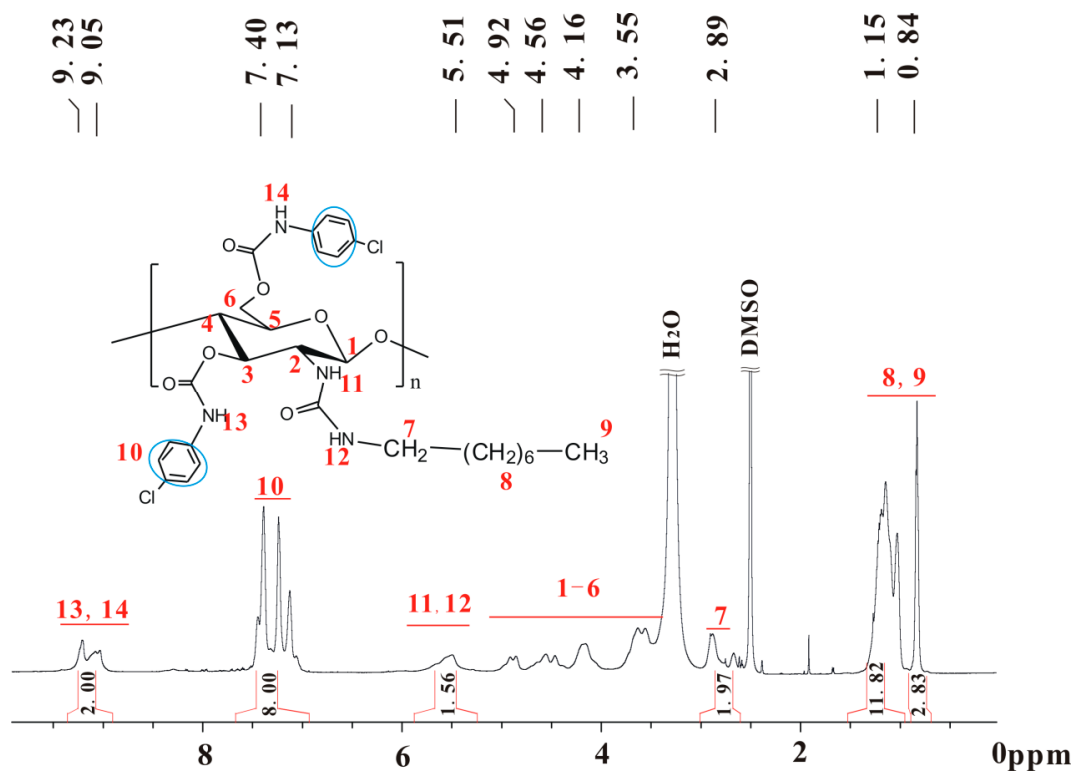
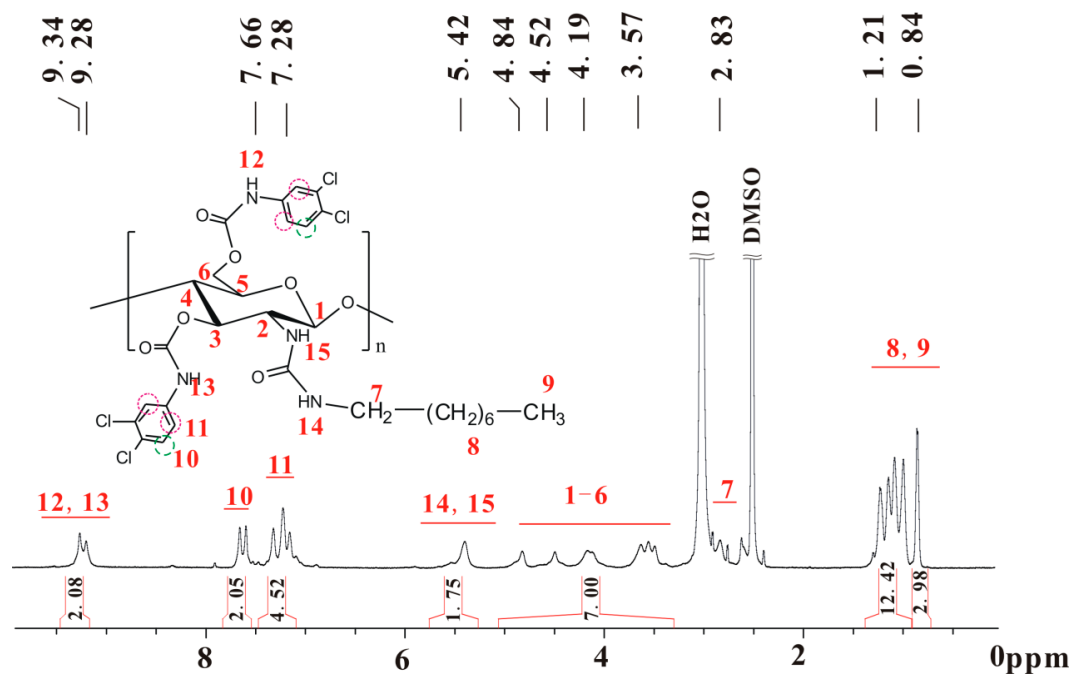
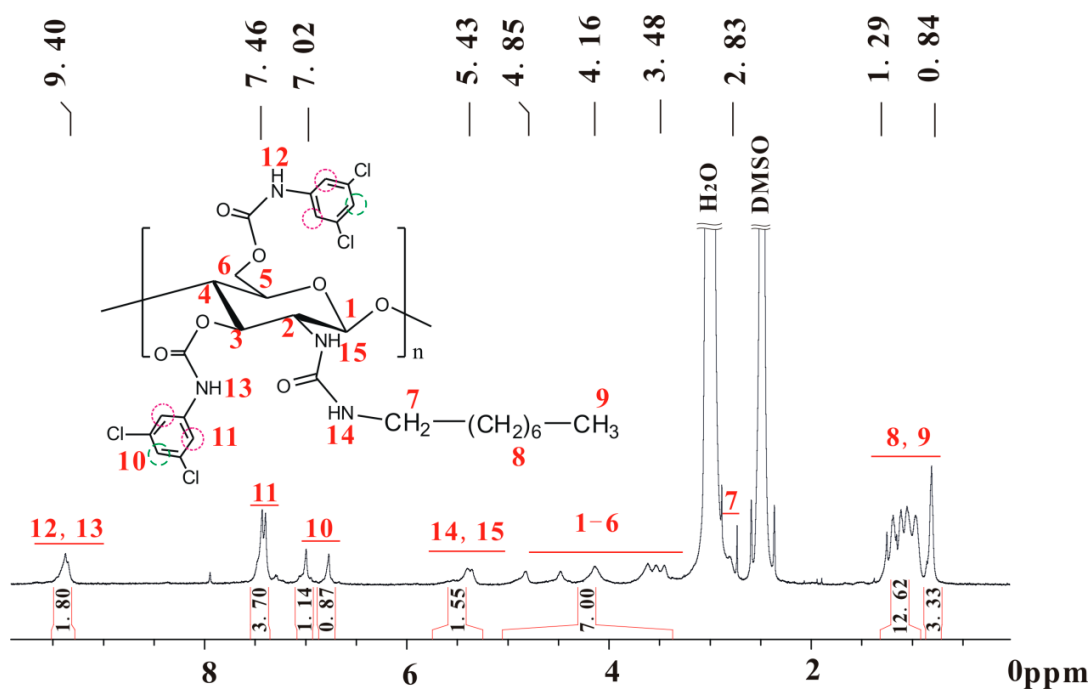
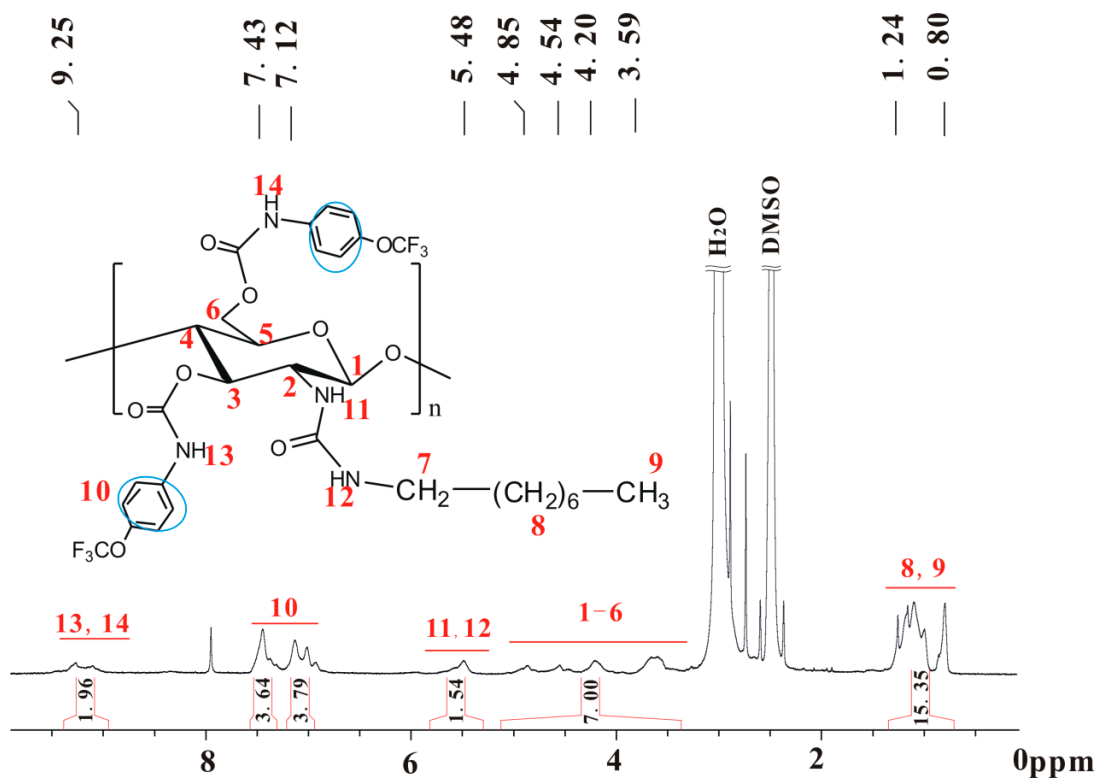


Figure S2. ¹H-NMR spectrum of CS 2.

Figure S3. ¹H-NMR spectrum of CS 3.Figure S4. ¹H-NMR spectrum of CS 4.

Figure S5. ¹H-NMR spectrum of CS 5.Figure S6. ¹H-NMR spectrum of CS 6.

2. IR Spectra of Chitosan Derivatives

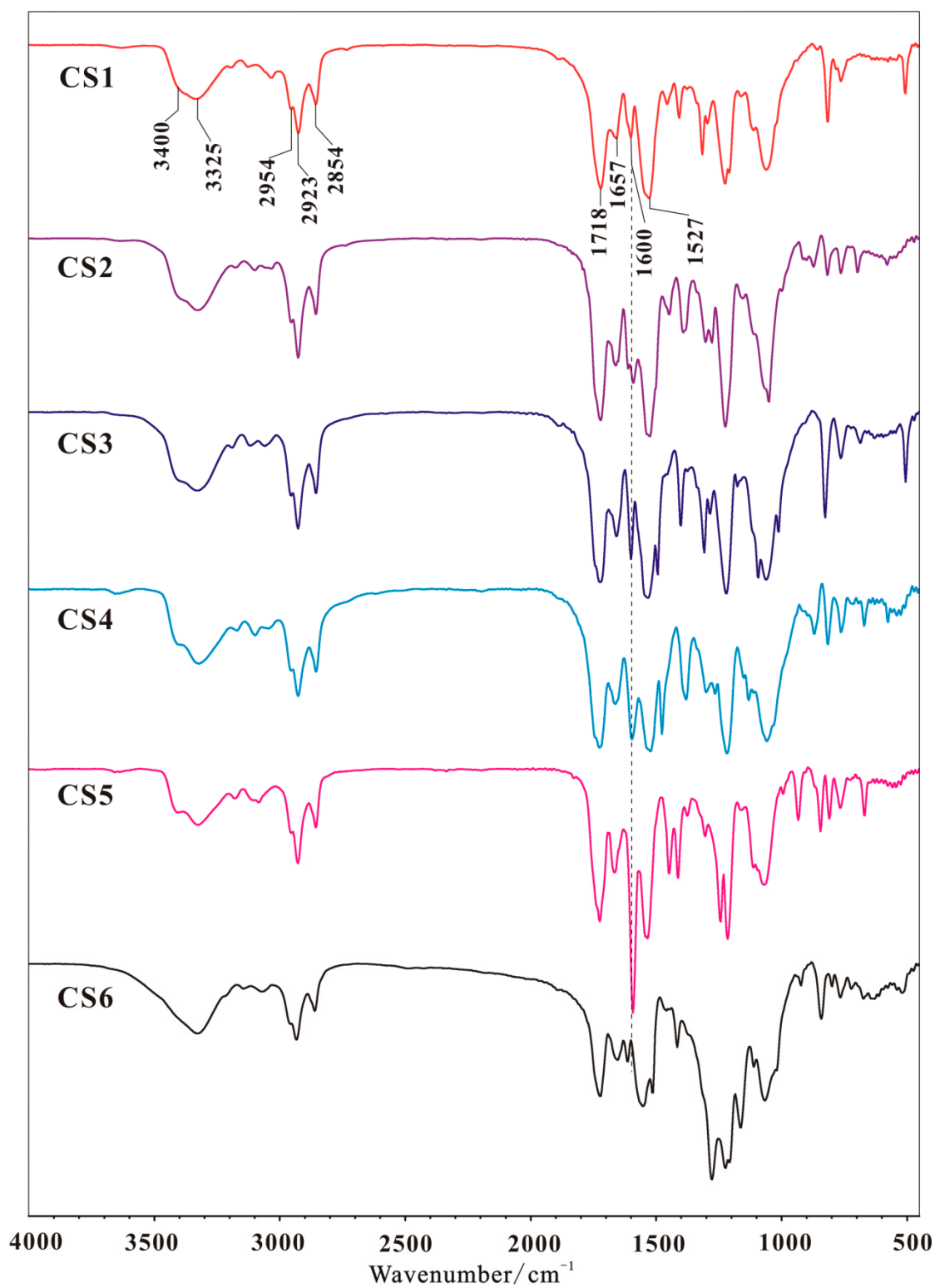


Figure S7. IR spectra of CSs 1–6.

3. Enantioseparation Evaluation of CSPs 1–6, ADMPC and CDMPC

Table S1. Enantioseparation evaluation of CSPs 1–6.

S.N.	CSP 1			CSP 2			CSP 3			CSP 4			CSP 5			CSP 6			M.P.
	k_1	α	R_s	k_1	α	R_s	k_1	α	R_s	k_1	α	R_s	k_1	α	R_s	k_1	α	R_s	
1	-0.27	1.09	0.46	-0.25	1.77	2.12	-0.29	1.75	3.21	-0.43	1.53	2.63	0.44	1.00	0.00	-0.21	1.60	1.67	A
	-0.24	1.11	0.54	-0.20	1.68	1.99	-0.27	1.55	2.45	-0.37	1.51	2.51	-0.34	1.37	1.54	-0.20	1.38	0.75	B
	-0.28	1.39	1.81	-0.22	1.50	1.60	-0.29	1.38	1.76	-0.39	1.33	1.64	-0.40	1.23	1.37	-0.23	1.18	0.51	C
2	1.56	1.00	0.00	1.10	1.00	0.00	0.90	1.00	0.00	0.88	1.00	0.00	0.92	1.00	0.00	0.66	1.00	0.00	A
	0.97	1.00	0.00	0.65	1.00	0.00	0.67	1.00	0.00	0.70	1.00	0.00	0.62	1.00	0.00	0.49	1.00	0.00	B
	0.93	1.00	0.00	0.61	1.00	0.00	0.64	1.00	0.00	0.76	1.00	0.00	0.67	1.00	0.00	0.63	1.00	0.00	C
3	+2.36	1.05	0.61	+2.33	1.08	0.58	-2.56	1.14	1.45	+4.70	1.04	0.55	-3.38	1.31	3.61	-2.03	1.16	1.04	A
	+1.80	1.08	1.04	1.64	1.00	0.00	+1.87	1.10	1.38	-2.95	1.00	0.00	-2.26	1.18	2.08	-1.29	1.29	2.96	B
	+1.77	1.26	2.44	1.52	1.00	0.00	+1.60	1.14	1.47	-2.52	1.00	0.00	-2.02	1.12	1.42	-1.23	1.31	2.79	C
4	-0.95	1.60	5.38	-1.13	1.36	2.73	-1.04	1.42	3.92	-2.67	1.28	2.68	-1.96	1.29	3.65	-0.71	1.48	2.32	A
	-0.82	1.72	6.61	-0.73	1.41	2.83	-0.77	1.42	3.81	-1.65	1.25	3.08	-1.26	1.27	3.01	-0.47	1.40	1.88	B
	-0.92	1.57	5.80	-0.70	1.32	2.29	-0.69	1.33	2.93	-1.35	1.21	2.29	-1.18	1.15	1.52	-0.49	1.35	2.06	C
5	+2.41	1.27	3.30	+3.27	1.19	1.50	+3.74	1.20	2.77	+8.57	1.11	1.68	+7.42	1.20	2.80	+3.30	1.29	2.36	A
	+1.35	1.25	2.84	+1.61	1.15	1.11	+2.06	1.18	2.21	+3.98	1.11	1.49	+3.57	1.16	2.25	+1.61	1.20	1.35	B
	+1.24	1.20	2.43	+1.35	1.05	0.46	+1.60	1.08	1.08	+2.81	1.07	0.99	+2.71	1.10	1.35	+1.51	1.10	1.01	C
6	-3.92	1.15	1.28	-3.28	0.16	0.46	-3.42	1.03	0.3	4.14	1.00	0.00	3.13	1.00	0.00	2.61	1.00	0.00	A
	-2.52	1.05	0.45	1.96	1.00	0.00	2.34	1.00	0.00	2.63	1.00	0.00	2.19	1.00	0.00	1.77	1.00	0.00	B
	2.18	1.00	0.00	1.64	1.00	0.00	1.86	1.00	0.00	2.22	1.00	0.00	1.75	1.00	0.00	1.69	1.00	0.00	C
7	0.72	1.00	0.00	0.44	1.00	0.00	0.35	1.00	0.00	0.38	1.00	0.00	0.41	1.00	0.00	0.28	1.00	0.00	A
	0.57	1.00	0.00	0.38	1.00	0.00	0.43	1.00	0.00	0.51	1.00	0.00	0.47	1.00	0.00	0.34	1.00	0.00	B
	0.63	1.00	0.00	0.46	1.00	0.00	0.48	1.00	0.00	0.59	1.00	0.00	0.50	1.00	0.00	0.46	1.00	0.00	C
8	^s 1.09	1.90	3.10	^s 1.01	1.24	1.15	^s 1.14	1.06	0.50	^s 3.26	1.07	0.74	^s 3.70	1.15	0.51	4.01	1.16	0.44	A
	^s 0.88	1.47	2.05	0.71	1.00	0.00	1.70	1.00	0.00	2.35	1.00	0.00	2.57	1.00	0.00	2.31	1.00	0.00	B
	^s 0.92	1.23	1.76	0.76	1.00	0.00	1.28	1.00	0.00	2.09	1.00	0.00	2.10	1.00	0.00	2.15	1.00	0.00	C
9	0.80	1.00	0.00	0.76	1.00	0.00	0.83	0.26	0.39	1.24	1.00	0.00	1.71	1.00	0.00	1.78	1.00	0.00	A
	0.51	1.00	0.00	0.46	1.00	0.00	1.08	1.00	0.00	0.91	1.00	0.00	0.91	1.00	0.00	3.07	1.00	0.00	B
	0.53	1.00	0.00	0.49	1.00	0.00	0.82	1.00	0.00	^R 0.96	1.05	0.54	0.89	1.00	0.00	2.38	1.00	0.00	C
10	2.75	1.00	0.00	^s 2.38	2.16	5.01	^s 3.41	1.54	2.39	^s 5.38	1.25	1.86	^s 3.73	1.46	2.28	^s 2.48	2.08	2.34	A
	^R 0.98	1.34	2.10	^s 0.97	1.35	2.13	^s 1.55	1.15	1.58	2.17	1.00	0.00	^s 1.38	1.24	2.20	^s 1.21	1.13	1.02	B
	^R 0.85	1.18	1.51	0.88	1.00	0.00	1.18	1.00	0.00	^R 1.79	1.09	0.99	1.26	1.00	0.00	1.20	1.00	0.00	C
11	1.36	1.00	0.00	2.20	1.00	0.00	3.27	1.00	0.00	^s 6.31	1.56	5.42	6.10	1.00	0.00	2.06	1.00	0.00	A
	0.72	1.00	0.00	1.14	1.00	0.00	1.66	1.00	0.00	^s 2.87	1.31	3.01	^s 2.63	1.14	1.53	1.03	1.00	0.00	B
	0.79	1.00	0.00	1.01	1.00	0.00	1.35	1.00	0.00	^s 2.24	1.26	2.37	^s 2.02	1.16	1.57	1.07	1.00	0.00	C

Table S1. Cont.

S.N.	CSP 1			CSP 2			CSP 3			CSP 4			CSP 5			CSP 6			M.P.
	k_1	α	R_s	k_1	α	R_s	k_1	α	R_s	k_1	α	R_s	k_1	α	R_s	k_1	α	R_s	
12	^R 9.97	1.39	2.78	^S 10.96	1.22	1.06	^S 7.80	1.20	1.19	^S 7.17	1.63	3.33	^S 3.87	1.71	3.97	^S 3.68	1.41	2.66	A
	^S 4.77	1.15	1.54	^S 4.22	1.61	3.03	^S 4.13	1.40	3.58	^S 3.19	2.00	4.10	^S 1.92	2.16	8.46	^S 1.71	1.46	3.15	B
	^S 3.40	1.09	1.12	^S 2.78	1.38	2.66	^S 2.64	1.26	2.69	^S 2.63	1.35	3.29	^S 1.45	1.61	5.09	^S 1.63	1.26	2.07	C
13	^S 2.81	1.09	0.88	1.88	1.00	0.00	1.88	1.00	0.00	^R 1.97	1.31	3.03	^R 1.56	1.44	3.37	1.45	1.00	0.00	A
	^R 1.56	1.06	0.56	^R 1.08	1.14	1.06	^R 1.25	1.06	0.55	^R 1.28	1.40	3.40	^R 0.93	1.98	9.80	0.91	1.00	0.00	B
	^R 1.37	1.12	1.31	^R 0.93	1.22	1.58	^R 1.03	1.07	0.61	^R 1.18	1.36	3.21	^R 0.88	2.36	6.92	0.92	1.00	0.00	C
14	⁺ 5.22	1.26	2.12	⁺ 4.57	1.17	1.16	⁺ 3.34	1.08	0.52	⁺ 6.58	1.07	0.58	⁺ 5.37	1.43	3.09	2.33	1.00	0.00	A
	⁺ 2.16	1.07	0.55	2.08	1.00	0.00	1.66	1.00	0.00	[−] 3.00	1.18	0.98	⁺ 2.42	1.28	1.23	1.20	1.00	0.00	B
	1.75	1.00	0.00	1.40	1.00	0.00	1.28	1.00	0.00	[−] 2.01	1.20	2.16	1.91	1.00	0.00	1.15	1.00	0.00	C
15	^S 13.99	1.11	0.65	7.61	1.00	0.00	^S 9.49	3.09	8.29	^S 9.25	2.90	7.82	^S 7.82	3.58	7.45	^S 8.05	3.79	8.50	A
	^S 2.93	1.04	0.40	^S 1.74	2.61	8.87	^S 2.56	1.97	6.74	^S 2.53	2.08	3.11	^S 1.87	2.48	10.65	^S 1.64	2.16	6.79	B
	^S 1.79	1.08	0.70	^S 1.26	1.40	3.03	^S 1.70	1.25	2.77	^S 2.20	1.09	1.02	^S 1.51	1.23	1.74	^S 1.51	1.21	2.09	C
16	^S 9.45	1.11	0.76	4.95	1.00	0.00	^S 5.79	2.94	7.61	^S 5.76	2.60	6.06	^S 6.24	2.89	6.62	^S 5.25	3.72	8.67	A
	1.99	1.00	0.00	^S 1.22	2.38	6.30	^S 1.72	1.73	4.07	^S 1.79	1.75	4.25	^S 1.44	2.08	6.33	^S 1.29	1.58	1.64	B
	1.24	1.00	0.00	^S 0.89	1.39	2.63	^S 1.22	1.19	1.87	^S 1.50	1.15	1.49	^S 1.08	1.31	2.01	^S 1.18	1.16	1.29	C
17	^R 2.41	1.35	3.27	^S 2.26	2.42	4.62	^S 3.65	1.52	2.87	^S 6.60	1.19	1.52	^S 4.88	1.34	2.63	^S 3.18	1.76	1.99	A
	^R 0.83	1.22	1.57	^S 1.05	1.39	2.72	^S 1.60	1.11	1.29	2.65	1.00	0.00	^S 1.65	1.14	2.09	^S 1.31	1.10	0.74	B
	^R 0.75	1.12	1.03	0.89	1.00	0.00	^R 1.17	1.06	0.58	^R 1.91	1.09	1.32	1.36	1.00	0.00	1.19	1.00	0.00	C
18	^R 14.77	1.48	4.39	^R 10.56	1.09	0.63	^S 11.33	1.15	1.20	^S 19.48	1.23	2.11	^S 16.57	1.15	1.65	^S 8.34	1.23	1.34	A
	^R 8.21	2.24	9.27	^R 5.54	1.28	1.96	^S 7.24	1.02	0.36	^S 11.58	1.03	0.42	^R 11.07	1.10	1.32	^S 4.48	1.14	0.86	B
	^R 6.53	1.93	8.37	^R 4.20	1.24	1.92	^S 4.88	1.08	0.96	^S 8.37	1.06	0.66	^R 8.05	1.14	1.66	^S 3.48	1.16	0.71	C
19	^S 15.59	1.16	0.92	^S 22.18	1.39	1.71	Retention time > 120min			Retention time > 120min			Retention time > 120min			Retention time > 120min			A
	^S 5.64	1.13	1.02	^S 7.51	1.39	2.14	^S 10.97	1.30	2.26	^S 15.23	1.32	3.31	^S 10.90	1.48	3.87	^S 6.35	1.42	3.26	B
	4.07	1.00	0.00	^S 5.34	1.21	1.51	^S 6.32	1.18	1.84	^S 8.88	1.12	1.30	^S 7.00	1.17	2.18	^S 4.31	1.22	2.04	C
20	Retention time > 120 min			⁺ 5.79	3.15	9.31	⁺ 4.26	2.54	6.76	⁺ 8.29	3.83	14.75	Retention time > 120min			⁺ 2.57	1.95	3.08	A
	⁺ 3.96	3.25	13.26	⁺ 2.40	2.32	7.35	⁺ 1.96	1.98	8.89	⁺ 3.49	2.45	10.69	⁺ 3.85	5.83	19.50	⁺ 1.33	1.22	2.20	B
	⁺ 2.74	2.37	10.12	⁺ 1.69	1.75	5.20	⁺ 1.56	1.42	4.40	⁺ 2.36	1.62	5.73	⁺ 2.44	3.93	16.41	⁺ 1.30	1.13	1.09	C

S.N.: serial number of the analytes; M.P. (mobile phase): A: *n*-hexane/isopropanol (90/10), B: *n*-hexane/ethanol (90/10), C: *n*-hexane/ethanol/methanol (90/5/5); flow rate: 1 ml/min; detection temperature: 25 °C. “+”, “−”, “R”, “S” and “⁺2S,3R”, “⁺4R,1S” at the superscript of k_1 refer to the optical rotation or configuration of the first-eluted enantiomer.

Table S2. Chiral separation results of ADMPC and CDMPC.

S.N.	ADMPC			CDMPC			M.P.
	k_1	α	R_s	k_1	α	R_s	
1	+0.65	1.51	1.68	+1.03	1.49	2.12	A
	+0.54	1.87	3.07	+0.70	1.64	2.62	B
	+0.66	1.79	3.79	+0.72	1.64	2.63	C
2	1.19	1.00	0.00	2.09	1.00	0.00	A
	+0.68	1.06	0.28	1.34	1.00	0.00	B
	0.98	1.00	0.00	1.24	1.00	0.00	C
3	3.99	1.00	0.00	+2.90	1.40	2.17	A
	+2.60	1.08	0.48	+2.06	1.33	1.93	B
	+2.76	1.10	0.85	+1.85	1.34	1.96	C
4	+1.24	1.20	1.37	-1.53	1.30	1.80	A
	+1.19	1.46	2.94	-1.15	1.21	1.14	B
	+1.43	1.79	5.00	-1.09	1.21	1.19	C
5	-2.95	1.06	0.48	+3.74	1.10	0.57	A
	1.71	1.00	0.00	1.90	1.00	0.00	B
	2.29	1.00	0.00	1.60	1.00	0.00	C
6	2.38	1.00	0.00	2.18	1.00	0.00	A
	1.78	1.00	0.00	1.60	1.00	0.00	B
	1.73	1.00	0.00	1.67	1.00	0.00	C
7	0.63	1.00	0.00	+0.95	1.12	0.42	A
	0.43	1.00	0.00	+0.65	1.13	0.22	B
	0.69	1.00	0.00	+0.57	1.13	0.38	C
8	1.65	1.00	0.00	2.49	1.00	0.00	A
	1.46	1.00	0.00	1.37	1.00	0.00	B
	1.95	1.00	0.00	1.23	1.00	0.00	C
9	^S 0.89	1.09	0.49	1.06	1.00	0.00	A
	0.75	1.00	0.00	0.75	1.00	0.00	B
	0.75	1.00	0.00	0.83	1.00	0.00	C
10	^R 2.66	1.42	2.42	^R 3.16	1.38	1.96	A
	^R 1.42	1.34	1.89	^R 1.35	1.26	1.30	B
	^R 1.13	1.28	1.60	^R 1.34	1.17	0.90	C
11	^{4R,1S} 2.69	2.36	4.81	2.97	1.00	0.00	A
	^{4R,1S} 1.65	1.89	3.73	^{4S,1R} 1.66	2.36	4.75	B
	^{4R,1S} 1.47	1.61	3.13	^{4S,1R} 1.86	2.06	4.36	C
12	^S 7.12	1.07	0.47	9.77	1.00	0.00	A
	^S 4.37	1.09	0.58	5.61	1.00	0.00	B
	^S 3.14	1.08	0.57	^S 3.78	1.08	0.51	C
13	^S 1.14	1.22	0.84	^R 1.62	1.34	1.60	A
	^S 0.58	1.12	0.32	^R 0.83	1.34	1.55	B
	0.97	1.00	0.00	^R 0.73	1.21	0.47	C
14	+2.42	1.57	2.45	5.71	1.00	0.00	A
	+1.37	2.98	6.28	1.75	1.00	0.00	B
	+1.72	2.87	6.98	1.36	1.00	0.00	C
15	^R 10.01	1.45	1.75	^S 11.74	1.20	0.96	A
	^R 1.97	1.28	1.33	^S 2.25	1.12	0.38	B
	^R 2.36	1.21	1.47	^S 2.50	1.10	0.35	C
16	^R 6.11	2.09	3.00	^R 7.61	1.25	0.39	A
	^R 1.32	1.41	1.60	^R 1.42	1.28	1.19	B
	^R 1.57	1.21	1.37	^R 1.05	1.20	0.89	C
17	^R 5.16	1.70	3.15	^R 4.52	1.18	1.01	A
	^R 1.22	1.46	2.04	^R 1.15	1.12	0.53	B
	^R 1.58	1.27	1.45	^R 1.10	1.16	0.54	C
18	^R 5.71	1.27	1.46	^S 9.85	1.09	0.96	A
	^R 3.23	1.11	0.77	4.89	1.00	0.00	B
	4.10	1.00	0.00	^R 3.82	1.10	0.45	C

Table S2. Cont.

S.N.	ADMPC			CDMPC			M.P.
	k_1	α	R_s	k_1	α	R_s	
19	Retention time>120 min			^R 17.56	1.28	0.43	A
	^R 10.37	1.61	2.57	^R 6.37	1.25	0.99	B
	^R 9.62	1.79	3.63	^R 3.94	1.15	0.54	C
20	^{2R,3S} 5.62	1.16	0.78	19.21	1.00	0.00	A
	^{2R,3S} 4.65	1.18	1.11	^{2R,3S} 9.47	1.29	1.81	B
	^{2R,3S} 3.81	1.17	1.16	^{2R,3S} 6.53	1.27	1.82	C

S.N.: serial number of the analytes; M.P.: mobile phase: A: *n*-hexane/isopropanol (90/10); B: *n*-hexane/ethanol (90/10); C: *n*-hexane/ethanol/methanol (90/5/5). “+”, “−”, “R”, “S”, “4R,1S”, “4S,1R” and “2R,3S” at the superscript of k_1 refer to the optical rotation or configuration of the first-eluted enantiomer. Flow rate: 1 mL/min. Detection temperature: 25 °C.

4. Enantioseparation Recovery of CSP 5

Table S3. Enantioseparation of CSP 5 under conditions I and V.

S.N.	Condition I			Condition V			M.P.
	k_1	α	R_s	k_1	α	R_s	
1	0.44	1.00	0.00	0.39	1.00	0.00	A
	0.34	1.37	1.54	0.31	1.28	1.40	B
	0.40	1.23	1.37	0.38	1.23	1.32	C
2	0.92	1.00	0.00	0.84	1.00	0.00	A
	0.62	1.00	0.00	0.57	1.00	0.00	B
	0.67	1.00	0.00	0.64	1.00	0.00	C
3	3.38	1.31	3.61	3.03	1.33	3.61	A
	2.26	1.18	2.08	2.06	1.22	2.25	B
	2.02	1.12	1.42	1.93	1.14	1.42	C
4	1.96	1.29	3.65	1.76	1.26	3.99	A
	1.26	1.27	3.01	1.16	1.29	2.99	B
	1.18	1.15	1.52	1.13	1.16	1.72	C
5	7.42	1.20	2.80	6.79	1.20	2.78	A
	3.57	1.16	2.25	3.29	1.17	2.25	B
	2.71	1.10	1.35	2.58	1.11	1.49	C
6	3.13	1.00	0.00	2.96	1.00	0.00	A
	2.19	1.00	0.00	2.13	1.00	0.00	B
	1.75	1.00	0.00	1.86	1.00	0.00	C
7	0.41	1.00	0.00	0.37	1.00	0.00	A
	0.47	1.00	0.00	0.42	1.00	0.00	B
	0.50	1.00	0.00	0.53	1.00	0.00	C
8	3.70	1.15	0.51	3.62	1.14	0.54	A
	2.57	1.00	0.00	2.44	1.00	0.00	B
	2.10	1.00	0.00	2.24	1.00	0.00	C
9	1.71	1.00	0.00	1.68	1.00	0.00	A
	0.91	1.00	0.00	0.95	1.00	0.00	B
	0.89	1.00	0.00	0.93	1.00	0.00	C
10	3.73	1.46	2.28	3.21	1.47	1.56	A
	1.38	1.24	2.20	1.37	1.20	2.14	B
	1.26	1.00	0.00	1.25	1.00	0.00	C
11	6.10	1.00	0.00	5.36	1.00	0.00	A
	2.63	1.14	1.53	2.30	1.16	1.60	B
	2.02	1.16	1.57	1.92	1.18	1.80	C
12	3.87	1.71	3.97	2.74	1.89	2.64	A
	1.92	2.16	8.46	1.79	2.20	8.71	B
	1.45	1.61	5.09	1.51	1.58	4.95	C

Table S3. Cont.

S.N.	Condition I			Condition V			M.P.
	k_1	α	R_s	k_1	α	R_s	
13	1.56	1.44	3.37	1.36	1.48	3.54	A
	0.93	1.98	9.80	0.87	2.03	8.52	B
	0.88	2.36	6.92	0.91	2.37	8.47	C
14	5.37	1.43	3.09	4.66	1.43	3.27	A
	2.42	1.28	1.23	2.38	1.24	2.07	B
	1.91	1.00	0.00	1.91	1.00	0.00	C
15	7.82	3.58	7.45	7.09	3.40	9.48	A
	1.87	2.48	10.65	1.81	2.49	9.06	B
	1.51	1.23	1.74	1.62	1.28	2.92	C
16	6.24	2.89	6.62	5.52	2.69	5.22	A
	1.44	2.08	6.33	1.39	2.06	6.75	B
	1.08	1.31	2.01	1.14	1.27	2.47	C
17	4.88	1.34	2.63	4.54	1.26	1.83	A
	1.65	1.14	2.09	1.66	1.09	1.16	B
	1.36	1.00	0.00	1.39	1.00	0.00	C
18	16.57	1.15	1.65	14.44	1.13	1.53	A
	11.07	1.10	1.32	9.56	1.11	1.28	B
	8.05	1.14	1.66	7.45	1.17	2.08	C
19	Retention time > 120 min			Retention time > 120 min			A
	10.90	1.48	3.87	10.19	1.45	3.72	B
	7.00	1.17	2.18	6.66	1.13	1.41	C
20	Retention time > 120 min			Retention time > 120 min			A
	3.85	5.83	19.50	3.54	5.76	19.07	B
	2.44	3.93	16.41	2.34	3.86	15.94	C

S.N.: serial number of the chiral compounds. M.P.: mobile phase: A: *n*-hexane/isopropanol (90/10); B: *n*-hexane/ethanol (90/10); C: *n*-hexane/ethanol/methanol (90/5/5). Flow rate: 1 mL/min; detection temperature: 25 °C. The conditions of CSP 5: I: after CSP 5 had been initially tested for its enantioseparation capability in *n*-hexane/isopropanol (90/10), *n*-hexane/ethanol 90/10, *n*-hexane/ethanol/methanol (90/5/5) and then placed aside for one month; V: after CSP 5 had been analyzed in condition IV (see Table 2) and then was placed aside for two months.