

**Preparation of novel bridged bis( $\beta$ -cyclodextrin) chiral  
stationary phase by thiol-ene click chemistry for enhanced  
enantioseparation in HPLC**

Ning Zhang, Siyu Guo and Bolin Gong\*

School of Chemistry and Chemical Engineering, North Minzu University, Yinchuan  
750021, China.

\*Prof. Bolin Gong

No. 204 Wenchang North Street, Xixia District, Yinchuan City, China

E-mail: [gongbolin@163.com](mailto:gongbolin@163.com)

### **Figure captions**

Fig. S1. The mass spectra of allyl-ureido- $\beta$ -CD (a) and bridged bis ( $\beta$ -CD) ligand (b).

Fig. S2. The  $^1\text{H}$ NMR spectrum of the bridged bis( $\beta$ -CD) ligand.

Fig. S3. The infrared spectra of the bridged bis( $\beta$ -CD) ligand (a) and HTC DP (b).

Fig. S4. The thermogravimetric analysis of HTC DP.

Fig. S5. Scanning electron microscopy (SEM) of HTC DP.

Fig. S6. Solid state  $^{13}\text{C}$  NMR of HTC DP. 105–65 ppm: C atoms on CD glucose unit.

Fig. S7. The chemical structures of 8 flavanones.

Fig. S8. The chemical structures of 8 triazole pesticides.

Fig. S9. The chemical structures of myclobutanil, ornidazole, voriconazole and lansoprazole.

Fig. S10. The chemical structures of 5 common chiral compounds.

Fig. S11. The effect of column temperature on the resolution, separation factor and retention factor of flavanones.

Fig. S12. The effect of column temperature on the resolution, separation factor and retention factor of triazole pesticides.

### **Table captions**

Table S1. The elemental analysis results of HTC DP and CDCSP.

Table S2. The effect of mobile phase type on 2-Hydroxyflavanone and 4-Hydroxyflavanone.

Table S3. Separation data of 2-hydroxyflavanone at different temperatures.

Table S4. The separation results and optimal separation conditions of myclobutanil, ornidazole, voriconazole and lansoprazole on HTC DP.

Table S5. Separation data of Hexaconazole at different temperatures.

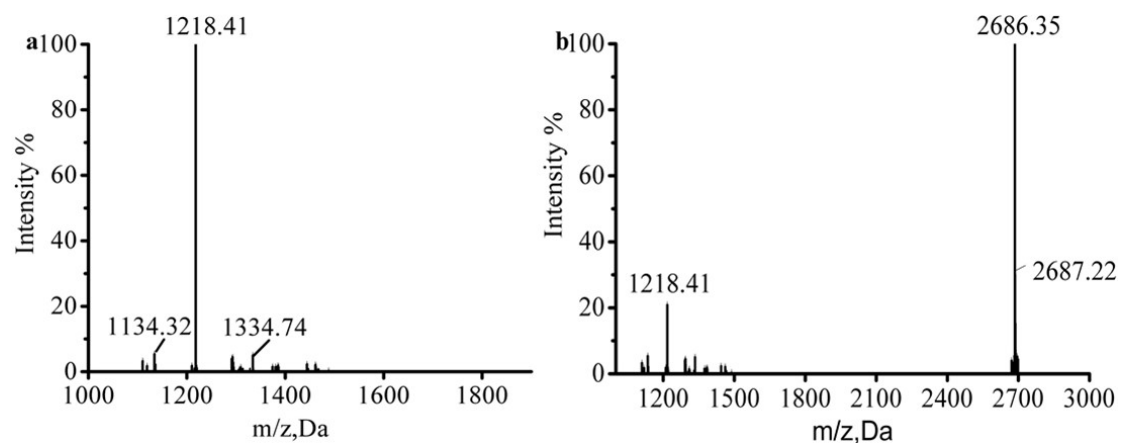


Fig. S1. The mass spectra of allyl-ureido- $\beta$ -CD (a) and bridged bis ( $\beta$ -CD) ligand (b)

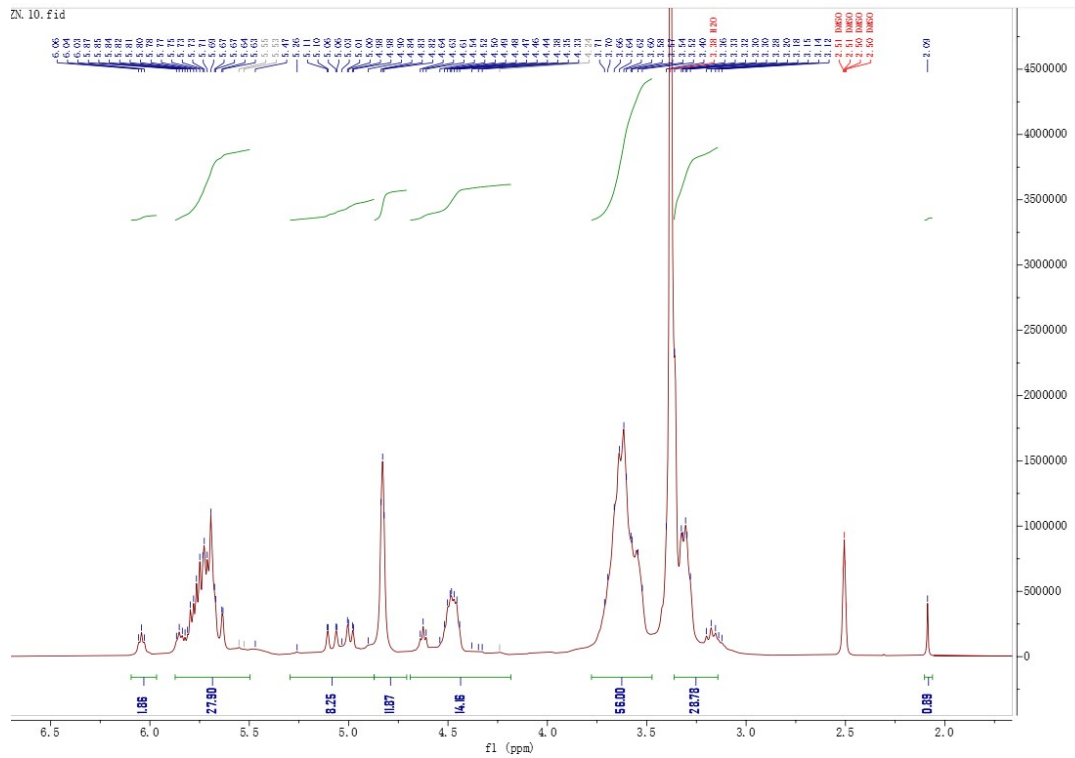


Fig. S2. The  $^1\text{H}$ NMR spectrum of the bridged bis( $\beta$ -CD) ligand

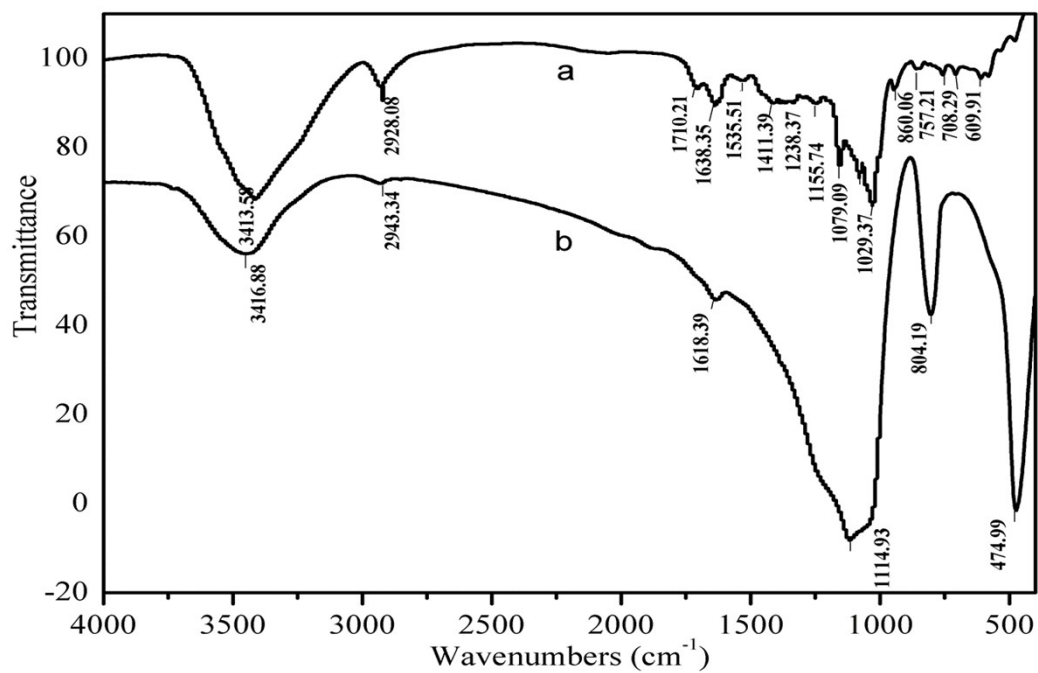


Fig. S3. The infrared spectra of the bridged bis( $\beta$ -CD) ligand (a) and HTCDP (b)

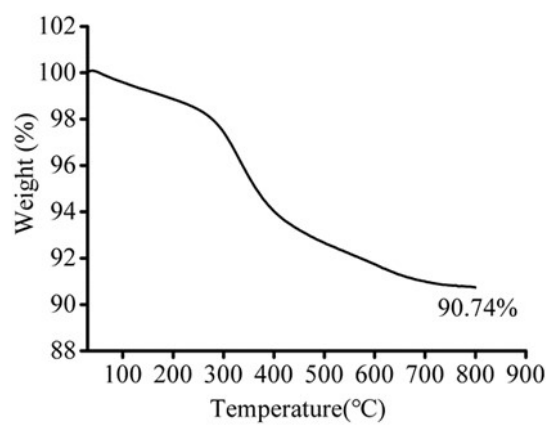


Fig. S4. The thermogravimetric analysis of HTC DP

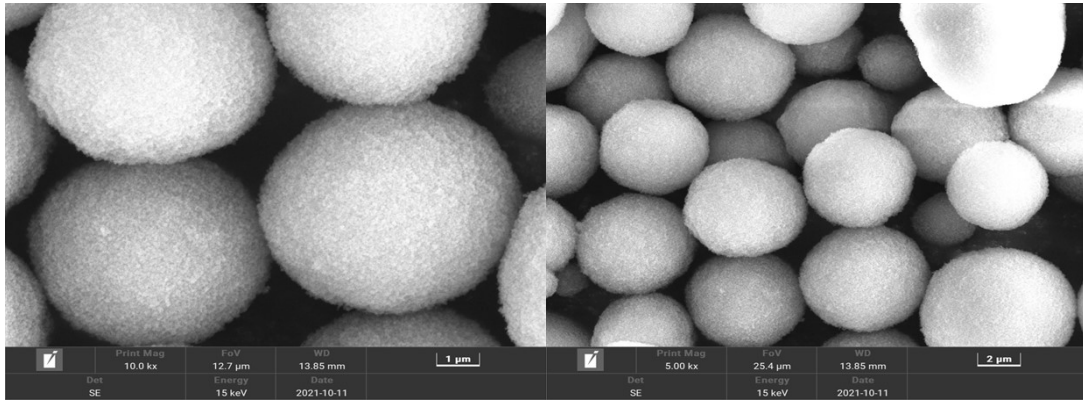


Fig. S5. Scanning electron microscopy (SEM) of HTCDP

:x20201023.1.fid  
P/TOSS double resonance

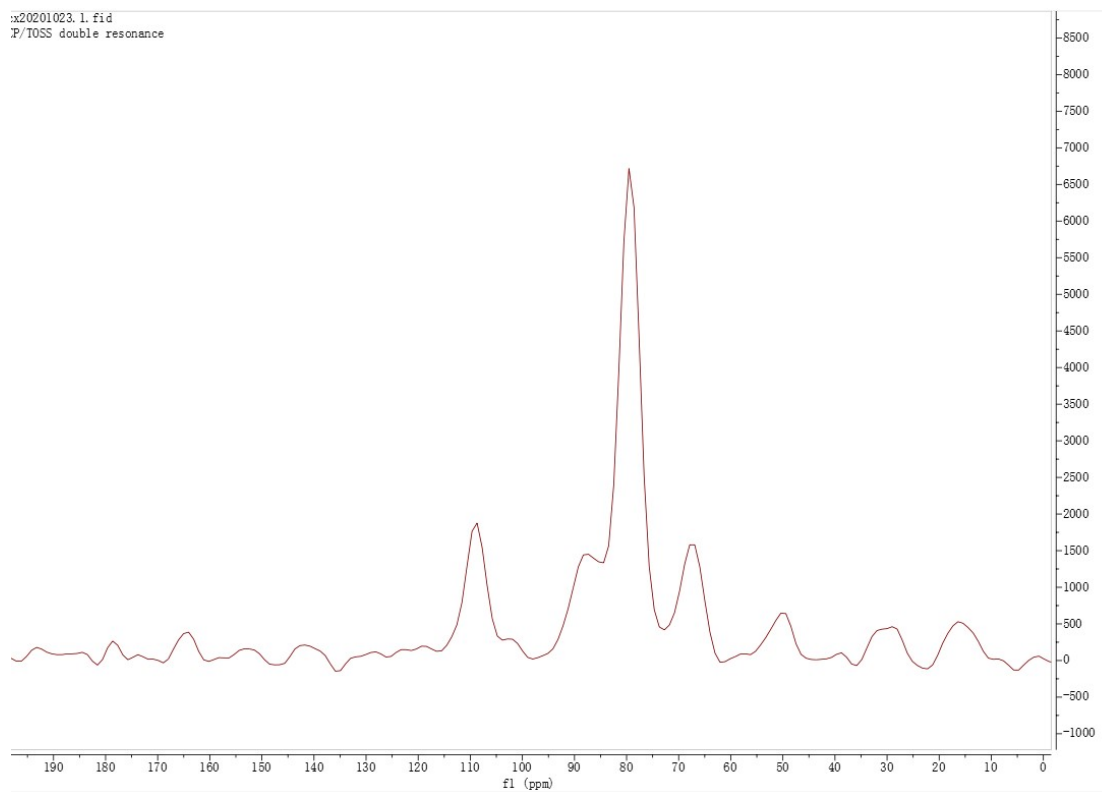


Fig. S6. Solid state <sup>13</sup>C NMR of HTCDP. 105–65 ppm: C  
atoms on CD glucose unit



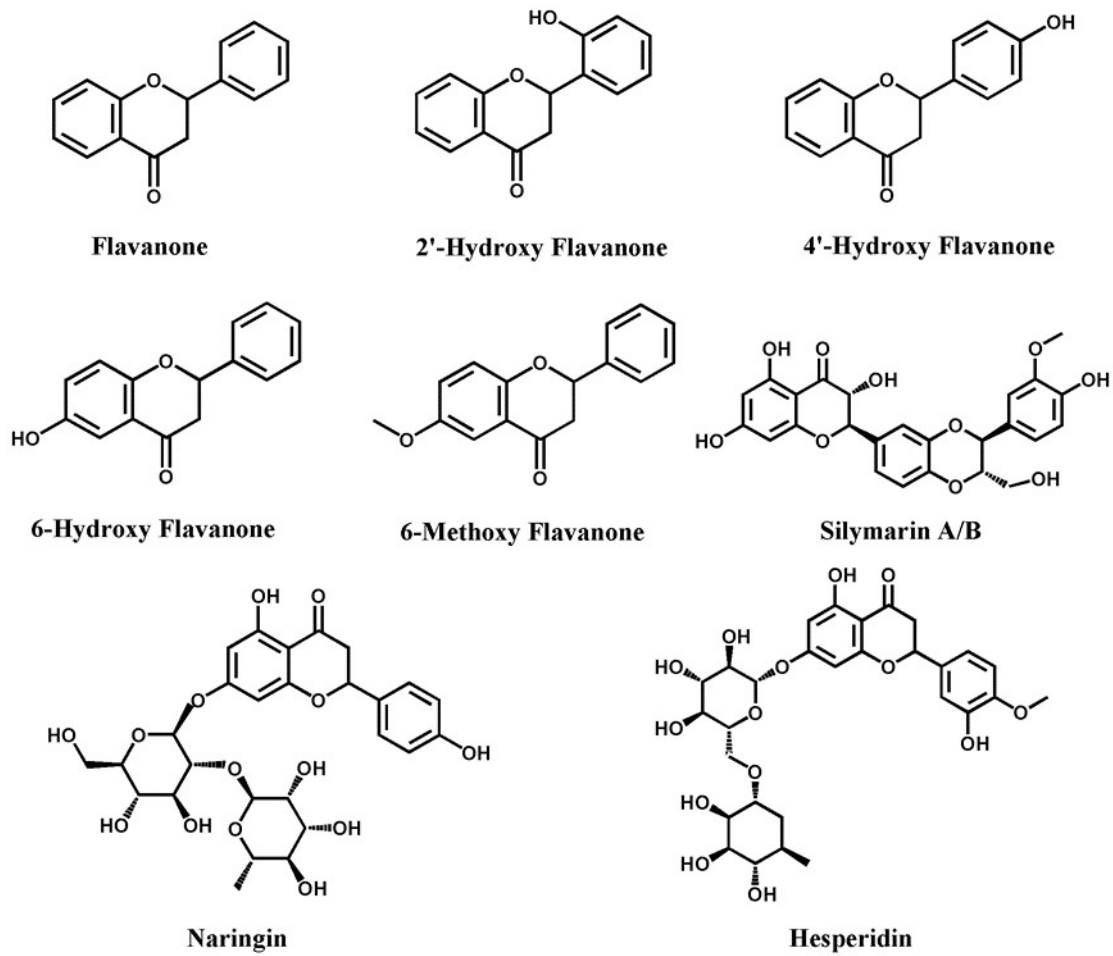
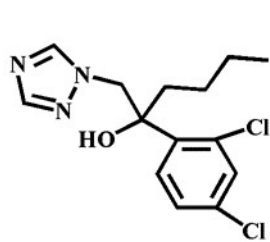
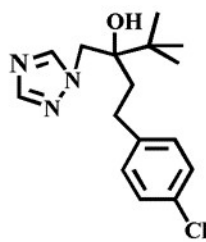


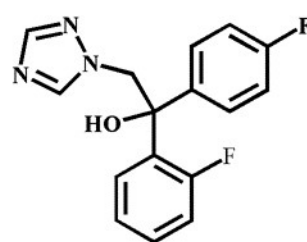
Fig. S7. The chemical structures of 8 common flavanones



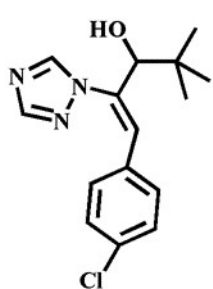
**Hexaconazole**



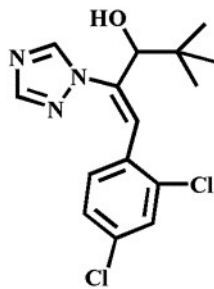
**Tebuconazole**



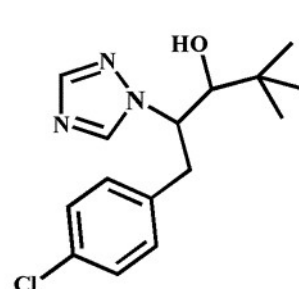
**Futriafol**



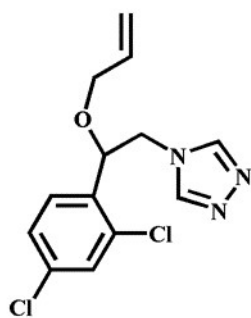
**Uniconazole**



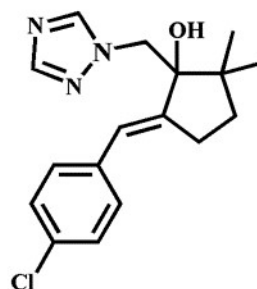
**Diniconazole**



**Paclbutrazol**

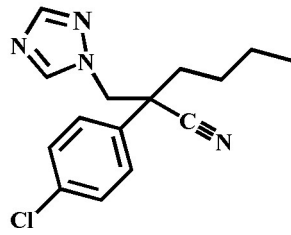


**Imazalil**

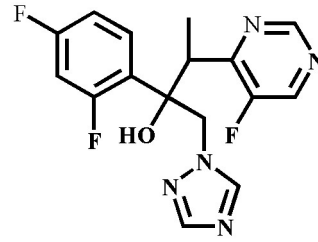


**Triticonazole**

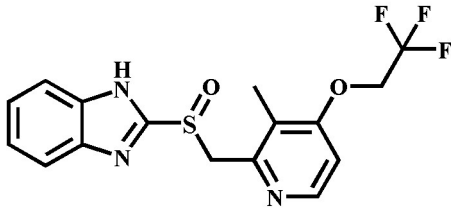
Fig. S8. The chemical structure of 8 triazole pesticides



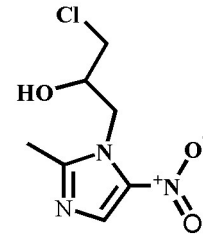
**Myclobutanil**



**Voriconazole**

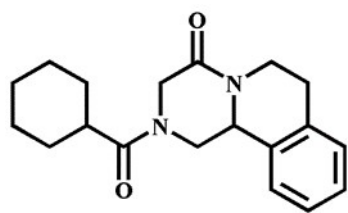


**Lansoprazole**

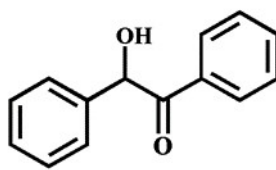


**Ornidazole**

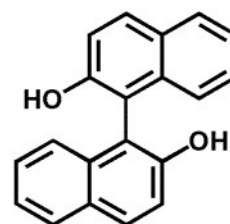
Fig. S9. The chemical structures of myclobutanil, ornidazole, voriconazole and lansoprazole



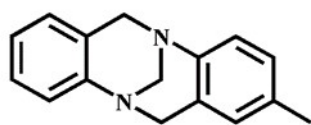
**Praziquantel**



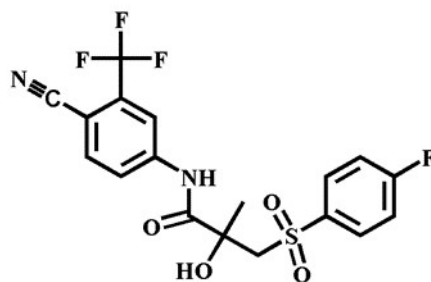
**Benoin**



**1-1'-Bi-2-naphthol**



**Troger base**



**Bicalutamide**

Fig. S10. The chemical structures of 5 common chiral compounds

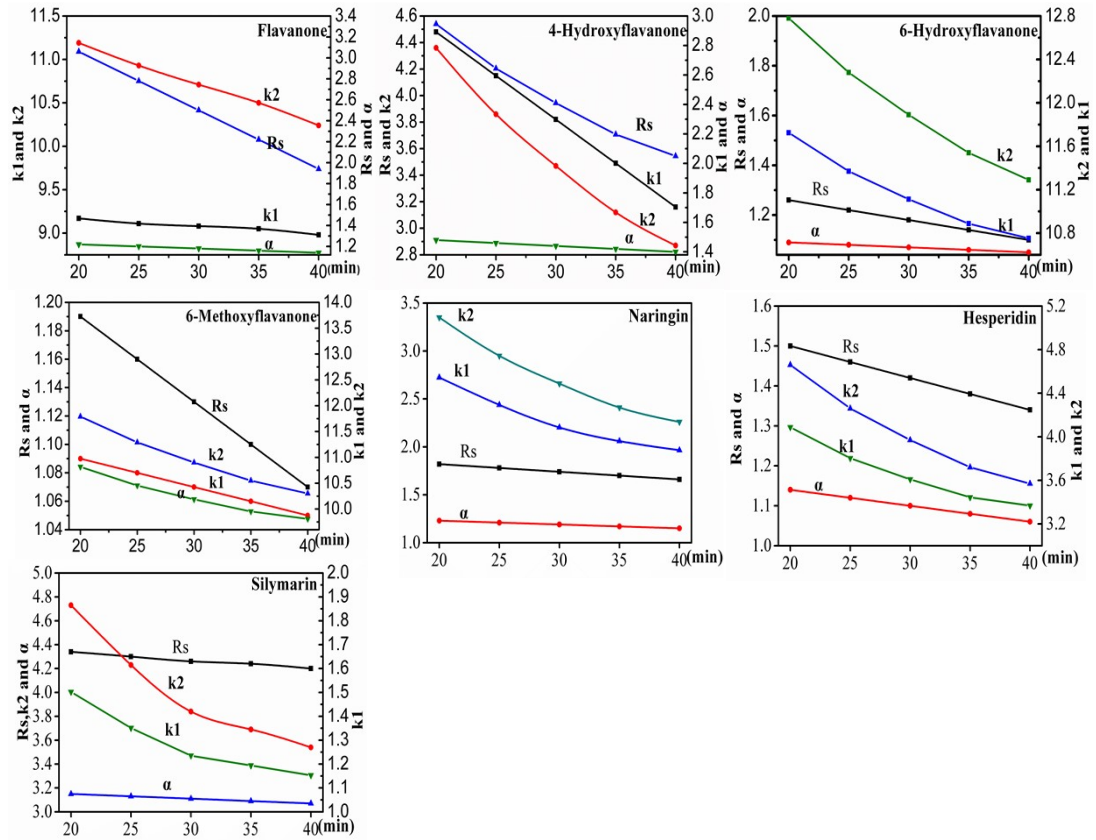


Fig. S11. The effect of column temperature on the resolution, separation factor and retention factor of flavanones

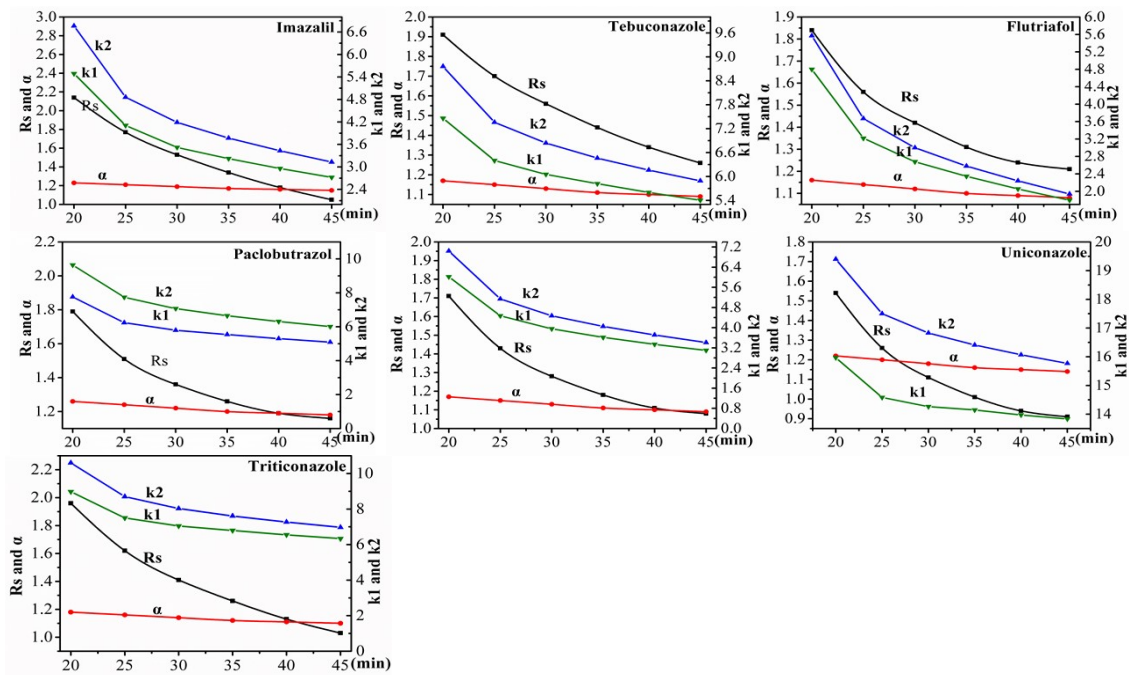


Fig. S12. The effect of column temperature on the resolution, separation factor and retention factor of triazole pesticides

Table S1. The elemental analysis results of HTC DP and CDCSP.

CSPs	C %	H %	N %	S %	Surface loading
HTCDP	3.23	0.46	0.24	0.12	0.45 $\mu\text{mol}/\text{m}^2$
CDCSP	1.60	0.28	0.38	/	0.51 $\mu\text{mol}/\text{m}^2$

Table S2. The effect of mobile phase type on 2-Hydroxyflavanone and 4-Hydroxyflavanone

Compounds	$k_1$	$k_2$	$\alpha$	$R_s$	Mobile phases(v/v)	Temp (°C)	CSPs
2-Hydroxy	2.11	2.96	1.40	3.39	ACN/0.1%FA (20/80)	20	HTCDP
flavanone	1.98	2.56	1.29	2.06	MeOH/0.1%FA (20/80)	20	HTCDP
4-Hydroxy	2.94	4.36	1.48	4.48	ACN/0.1%FA (20/80)	20	HTCDP
flavanone	2.76	3.61	1.31	2.68	MeOH/0.1%FA (20/80)	20	HTCDP



Table S3. Separation data of 2-hydroxyflavanone at different temperatures

	Rs	k <sub>1</sub>	k <sub>2</sub>	$\alpha$
20 °C	3.89	2.11	2.96	1.40
25 °C	3.56	1.78	2.46	1.38
30 °C	3.24	1.52	2.07	1.36
35 °C	2.91	1.28	1.72	1.34
40 °C	2.58	1.11	1.47	1.32

Table S4. The separation results and optimal separation conditions of myclobutanil, ornidazole, voriconazole and lansoprazole on HTCDP

No.	Compounds	k <sub>1</sub>	k <sub>2</sub>	$\alpha$	Rs	Mobile phases(v/v)	Temp (°C)	CSPs
1	Myclobutanil	4.38	4.75	1.08	<0.5	ACN/H <sub>2</sub> O (10/90)	20	HTCDP
2	Ornidazole	0.29	/	/	/	ACN/H <sub>2</sub> O (10/90)	20	HTCDP
3	Voriconazole	2.14	/	/	/	ACN/H <sub>2</sub> O (10/90)	20	HTCDP
4	Lansoprazole	2.21	/	/	/	ACN/H <sub>2</sub> O (15/85)	20	HTCDP

Note: k', retention factor;  $\alpha$ , separation factor; Rs, resolution; Temp, temperature (°C); CSPs, chiral stationary phase; HTCDP, a novel bridged bis( $\beta$ -cyclodextrin) chiral stationary phase; CDCSP, native  $\beta$ -cyclodextrin chiral stationary phase; /, no separation.

Table S5. Separation data of Hexaconazole at different temperatures

	Rs	k <sub>1</sub>	k <sub>2</sub>	$\alpha$
20°C	2.19	4.48	5.42	1.21
25°C	1.82	2.96	3.52	1.19
30°C	1.58	2.43	2.85	1.17
35°C	1.39	2.11	2.43	1.15
40°C	1.23	1.83	2.09	1.14
45°C	1.10	1.59	1.79	1.13