

“One-pot” click access to triazole bridged cyclodextrin chiral phases for differentiation of clopidogrel enantiomers

Qing Kang, Xiaobin Yao, Lifang Zhang, Zhihua Wu and Yong Wang*

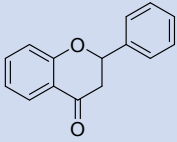
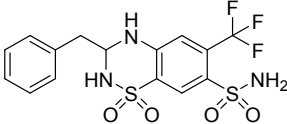
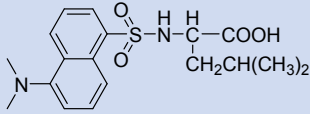
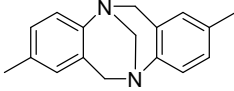
Department of Chemistry, School of Sciences, Tianjin University, Tianjin 300072, China.
Collaborative Innovation Center of Chemical Science and Engineering (Tianjin), Tianjin 300072, China

Synthesis and characterization of *N*-3-(triethoxysilyl)propyl-2-propynamide

To a solution of 3-aminopropyltriethoxysilane (APTES) (1.2 g, 5.4 mmol) and propiolic acid (0.44 g, 5.96 mmol) in dry CH₂Cl₂ (10 mL) was added *N,N'*-dicyclohexylcarbodiimide (DCC) (1.22 g, 5.95 mmol). The reaction mixture was kept at 0 °C for 10 min and then 25 °C for 1 hr. The reaction mixture was filtered and the filtration was concentrated followed by coevaporation with toluene (twice) affording *N*-3-(triethoxysilyl)propyl-2-propynamide as a light yellow syrup.

¹H NMR (400 MHz, CDCl₃): δ=7.29 (1H, NH), 3.78 (6H, CH₂O), 3.21 (2H, CH₂NH), 2.90 (1H, C≡CH), 1.59 (2H, CH₂CH₂NH), 1.18 (9H, OCH₂CH₃), 0.62 (2H, CH₂Si); ¹³C NMR (75 MHz, CDCl₃): δ=161.2, 76.9, 72.8, 58.0, 41.5, 21.8, 17.4, 7.2. FT-IR shows a strong absorption around 2120 cm⁻¹ (C≡CH).

Table S1 Optimal enantioseparation of flavanone, bendroflumethiazide, dansyl-DL-Leucine and Tröger's base on **CSP1-5**.

Analytes	α	R_s	CSP	Conditions
Flavanone 	1.36	2.76	CSP1	ACN/H ₂ O 30/70
	1.19	1.96	CSP2	MeOH/H ₂ O 30/70
	1.79	4.23	CSP3	MeOH/H ₂ O 40/60
	1.13	1.25	CSP4	ACN/H ₂ O 30/70
	1.37	4.41	CSP5	ACN/H ₂ O 30/70
Bendroflumethiazide 	1.01	<0.3	CSP1	MeOH/H ₂ O 20/80
	1.00	-	CSP2	MeOH/H ₂ O 20/80
	1.67	3.55	CSP3	MeOH/H ₂ O 30/70
	1.00	-	CSP4	MeOH/H ₂ O 20/80
	1.01	0.7	CSP5	MeOH/H ₂ O 20/80
Dansyl-DL-leucine 	1.98	5.03	CSP1	MeOH/Buffer 50/50
	1.00	-	CSP2	MeOH/Buffer 50/50
	1.00	-	CSP3	MeOH/Buffer 50/50
	1.23	1.54	CSP4	MeOH/Buffer 50/50
	2.02	7.69	CSP5	MeOH/Buffer 50/50
Tröger's base 	1.10	1.51	CSP1	MeOH/H ₂ O 35/65
	1.00	-	CSP2	MeOH/H ₂ O 35/65
	1.00	-	CSP3	MeOH/H ₂ O 35/65
	1.19	1.69	CSP4	MeOH/H ₂ O 35/65
	1.12	1.88	CSP5	MeOH/H ₂ O 35/65

Buffer: 1% Triethylammonium acetate (pH=4.1); flow rate: 0.8 mL·min⁻¹.

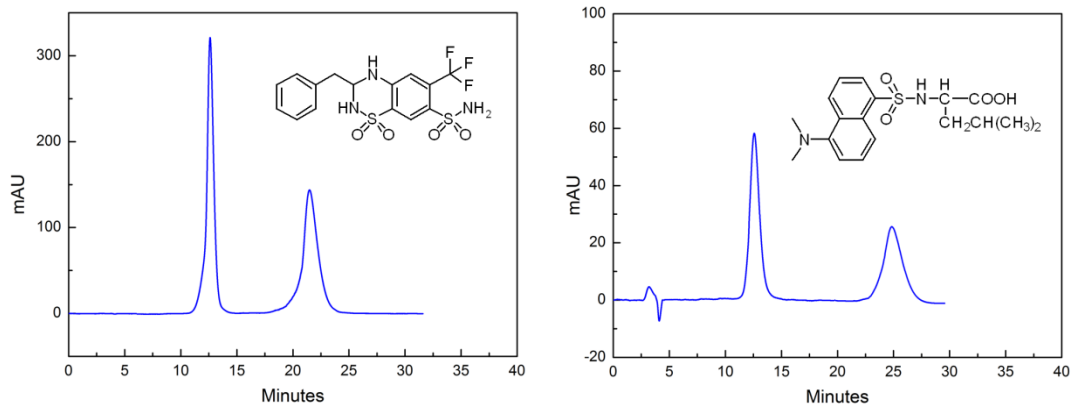


Fig.S1 Typical chromatograms on the prepared CDCSPs. Left: CSP3, MeOH/H₂O 30/70; Right: CSP5, MeOH/Buffer 50/50; Other conditions: see Table S1.