

**Poly(vinyl alcohol)-cationic cellulose copolymer encapsulated SiO₂ stationary phase for
hydrophilic interaction liquid chromatography**

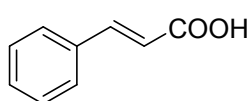
Yahui Peng, Feifang Zhang, Xiao Pan, Yanjie Hou, Bingcheng Yang*

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China*

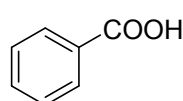
*Corresponding Authors: Tel +86-21-64250627; Email, bcyang@ecust.edu.cn (B. Yang).

Experimental

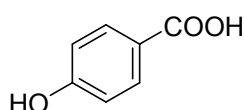
Elemental analysis was studied on a Varil EL III Element Analyzer (ELEMENTAR Analysen system GmbH, Germany). X-ray photoelectron spectroscopy (XPS) was obtained with an Escalab 250Xi photoelectron spectrometer (Thermo Scientific, NY, USA). Zeta-potential analysis was performed on a Malvern Zetasizer Nano ZS90 (Malvern, UK).



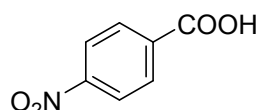
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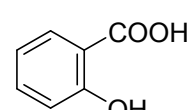
Benzoic acid



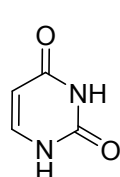
4-Hydroxybenzoic acid



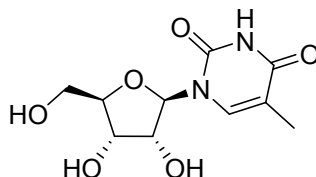
4-Nitrobenzoic acid



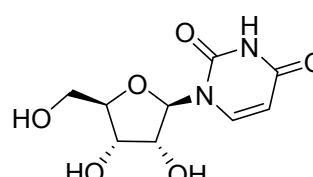
Salicylic acid



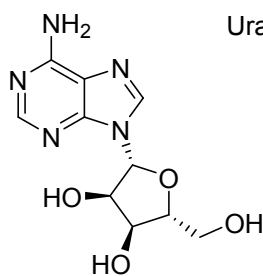
Uracil



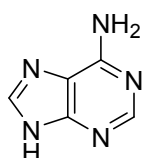
5-Methyl uridine



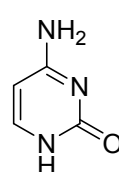
Uridine



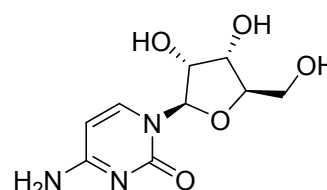
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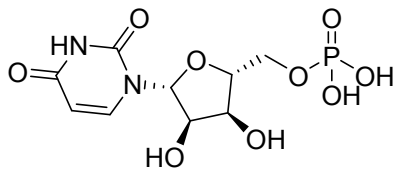
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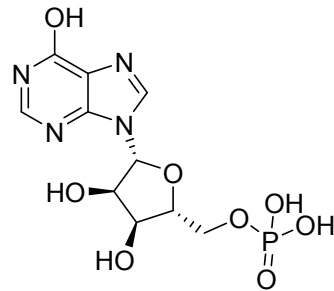
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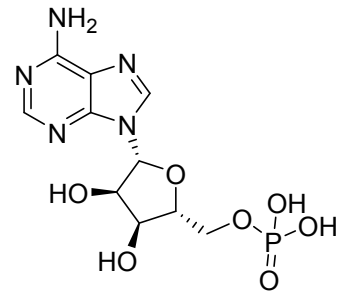
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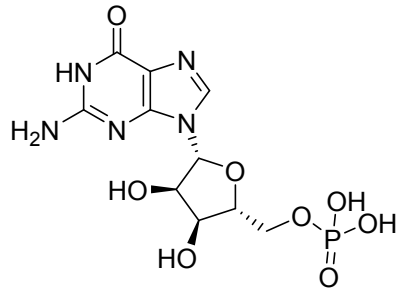
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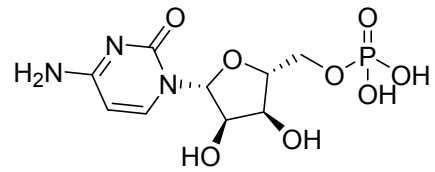
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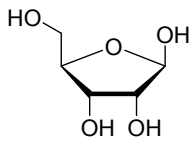
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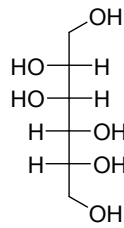
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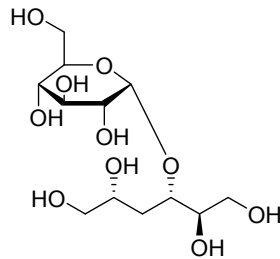
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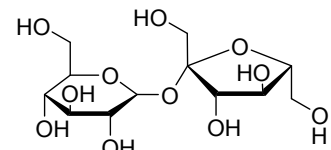
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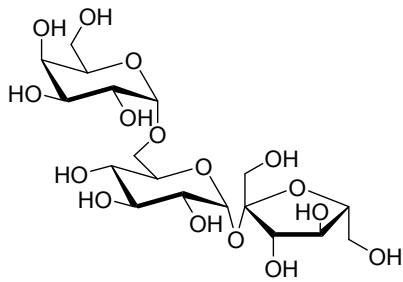
Mannitol



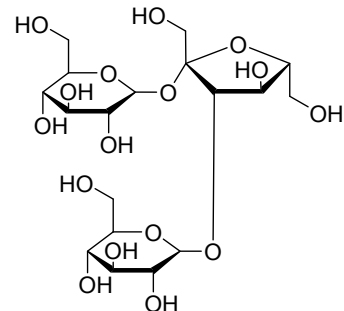
Maltitol



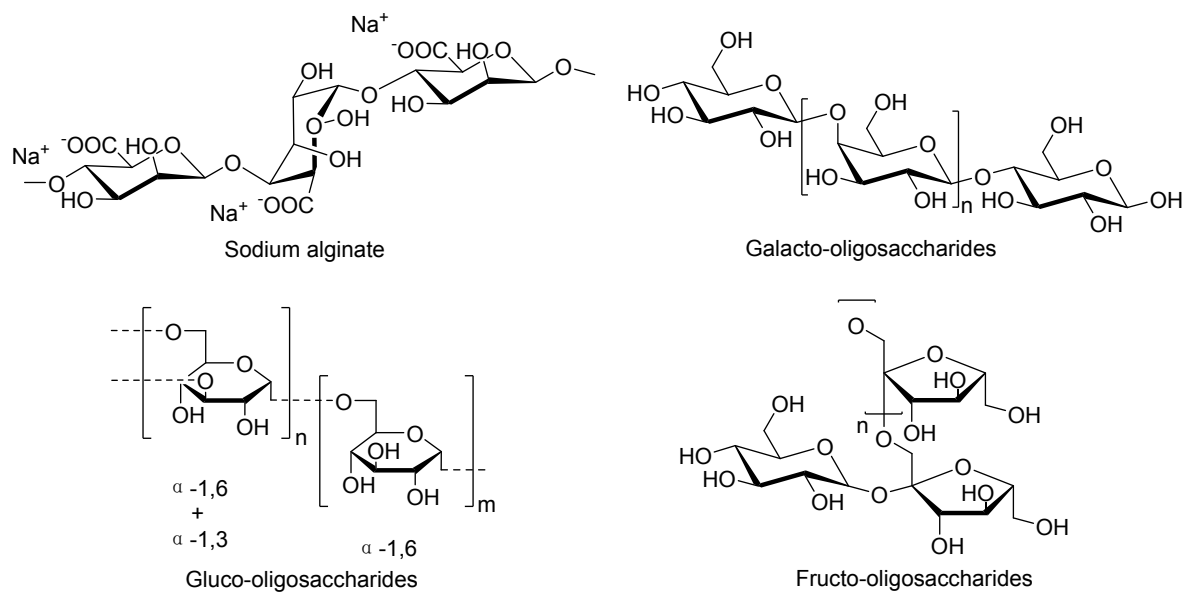
Sucrose



Raffinose



Melezitose

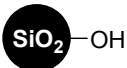
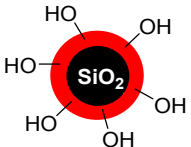
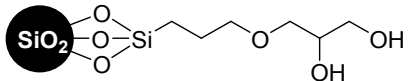
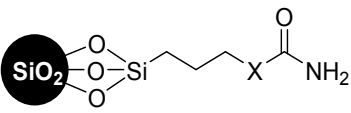
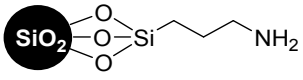
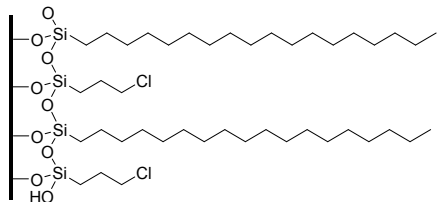


SI-Scheme 1 Chemical structures of the analytes chosen for chromatographic evaluation

1.1 Zeta-potential measurement

Into 1 mL of 1.67 mg/mL bare silica or PVA-CC-Sil dispersion in water was added 2 mL of 100 mM HCOONH_4 at different pH values. The mixture was then diluted with deionized water to a final volume of 10 mL and subjected to sonication for 1 min before test.

SI-Table 1 Chemical information of the domestic columns used for comparison

Column name	Bonded phase	Chemical Structure
Bare silica	none	
PVA-Sil	diol	
ACCHROM Unitary Diol	diol	
ACCHROM XAmide	amide	
ACCHROM Unitary NH2	amine	
ACCHROM SAX	amine	
ACCHROM Click XIon	ZIC	
ACCHROM XAqua C18	C18	
ACCHROM Xcharge C18	C18	

SI-Table 2 Elemental analysis of PVA-Sil and PVA-CC-Sils

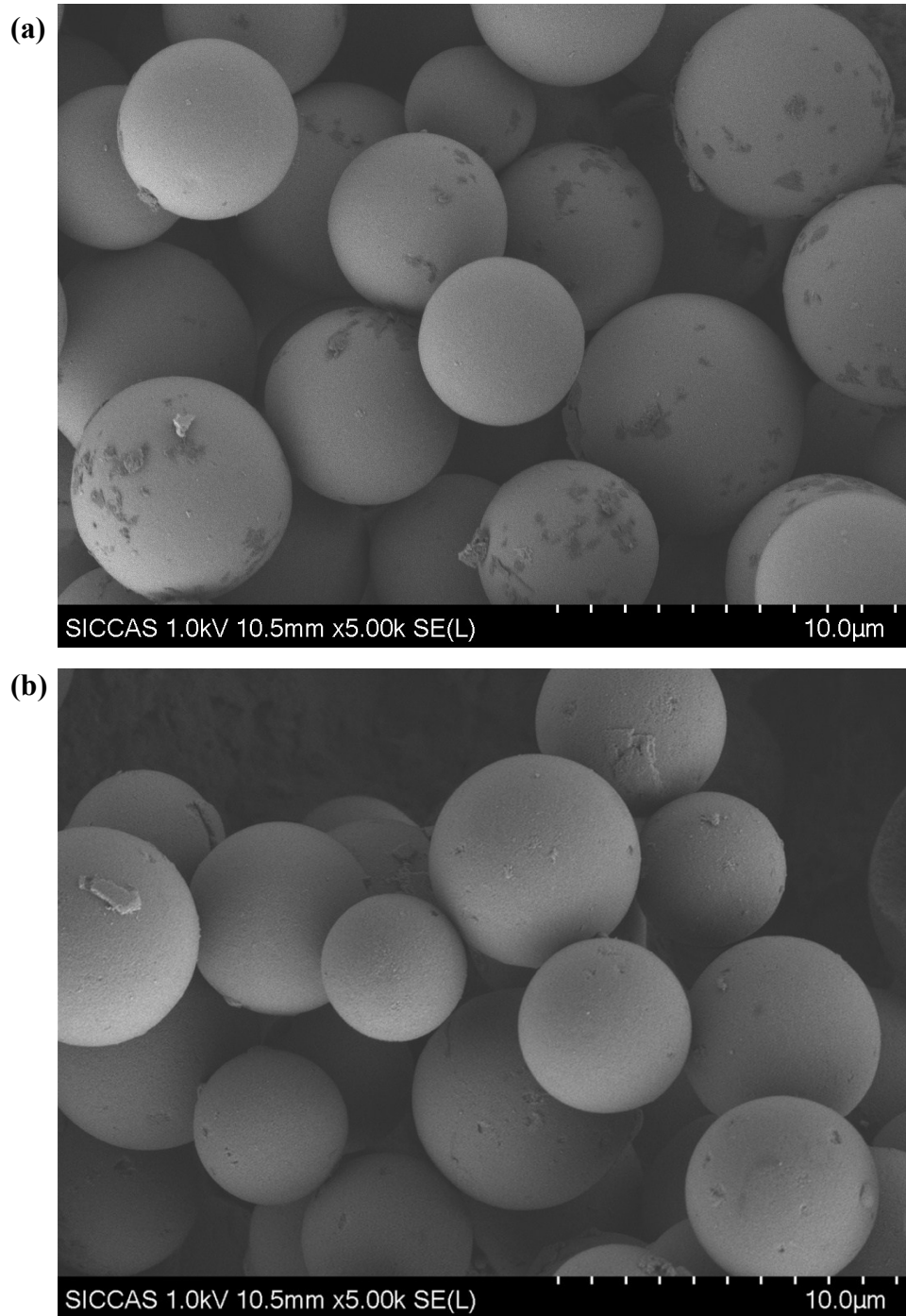
Stationary phases	C%	N%	H%	Loading of cationic cellulose (mg/g)
Bare silica gel	0.55	-	1.26	-
PVA	54.55	-	9.09	-
Cationic cellulose	37.87	2.06	6.43	-
PVA-CC-Sil	8.85	0.11	2.17	53.88

SI-Table 3 Surface and pore analysis of bare silica gel and PVA-CC-Sil

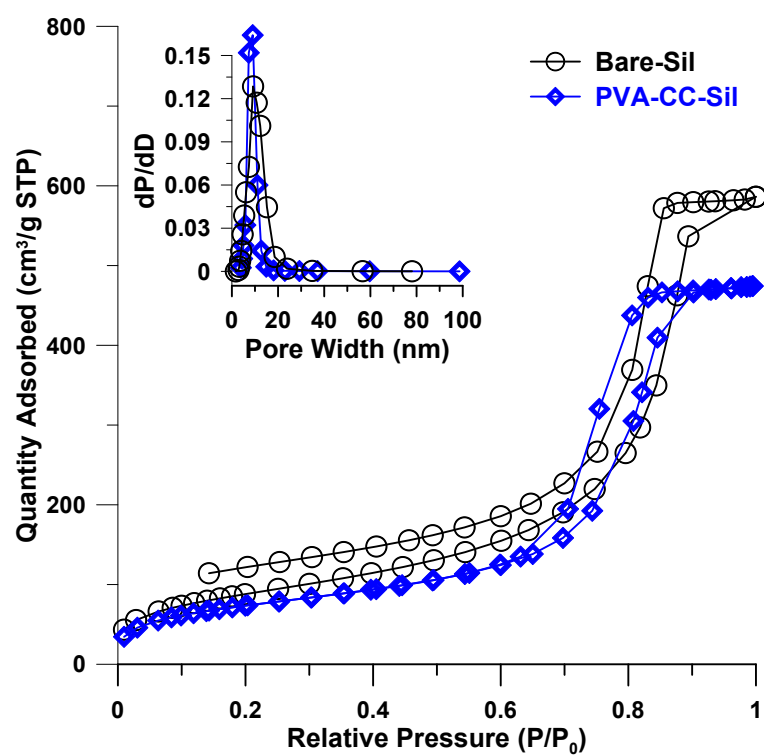
Stationary phases	BET Surface area	BJH Adsorption	BJH Adsorption
	(m ² /g)	Pore Volume (cm ³ /g)	Pore Diameter (Å)
Bare silica gel	324	0.92	97
PVA-CC-Sil	271	0.74	92

SI-Table 4 van't Hoff equations of five nucleosides and nucleic acid bases on PVA-CC-Sil

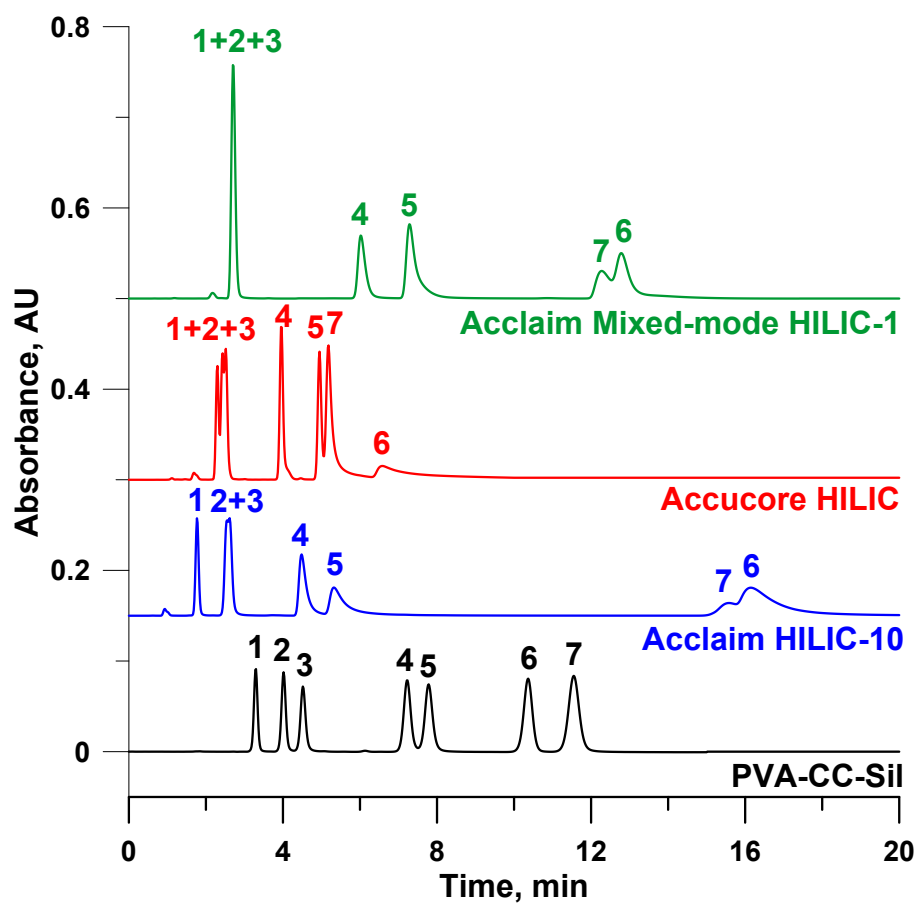
Analyte	$-\frac{\Delta H^0}{R}$	$\frac{\Delta S^0}{R} + \ln \Phi$	Retention enthalpy (kJ/mol)	r^2
Uracil	495.274	-1.299	-4.1	0.990
5-Methyluridine	767.292	-1.908	-6.4	0.999
Uridine	768.474	-1.743	-6.4	0.998
Adenine	848.544	-1.271	-7.1	0.997
Cytosine	691.723	-0.426	-5.8	0.999



SI-Fig. 1 Scanning electron micrographs of (a) bare silica gel and (b) PVA-CC-Sil

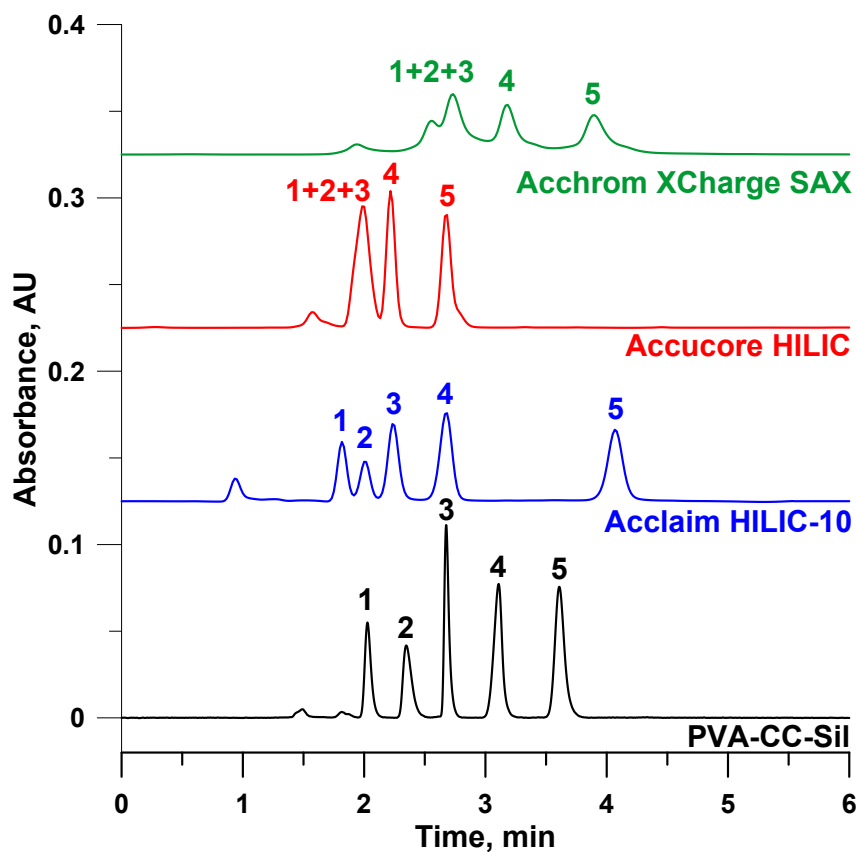


SI-Fig. 2 N₂ adsorption-desorption isotherms and BJH-adsorption pore size distribution of bare silica gel and PVA-CC-Sil.



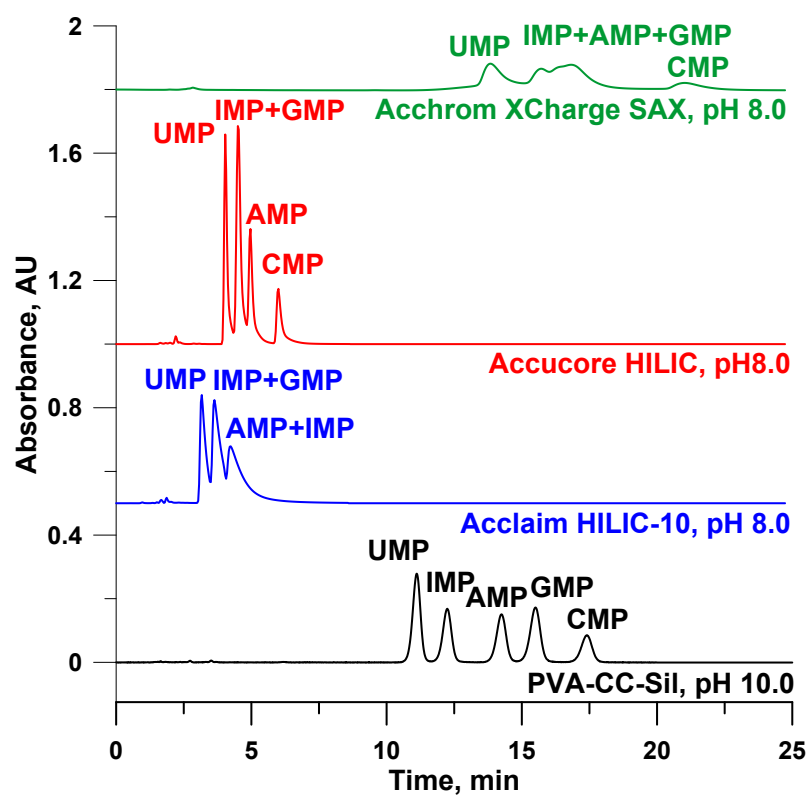
SI-Fig. 3 Comparative separation chromatograms of nucleosides on PVA-CC-Sil, Acclaim HILIC-10, Accucore HILIC and Acclaim Mixed-mode HILIC-1.

Conditions: 90% ACN/10% H₂O for PVA-CC-Sil and Accucore HILIC; 95% ACN/5% H₂O for Acclaim HILIC-10 and Acclaim Mixed-mode HILIC-1; other conditions same as Fig. 3b.



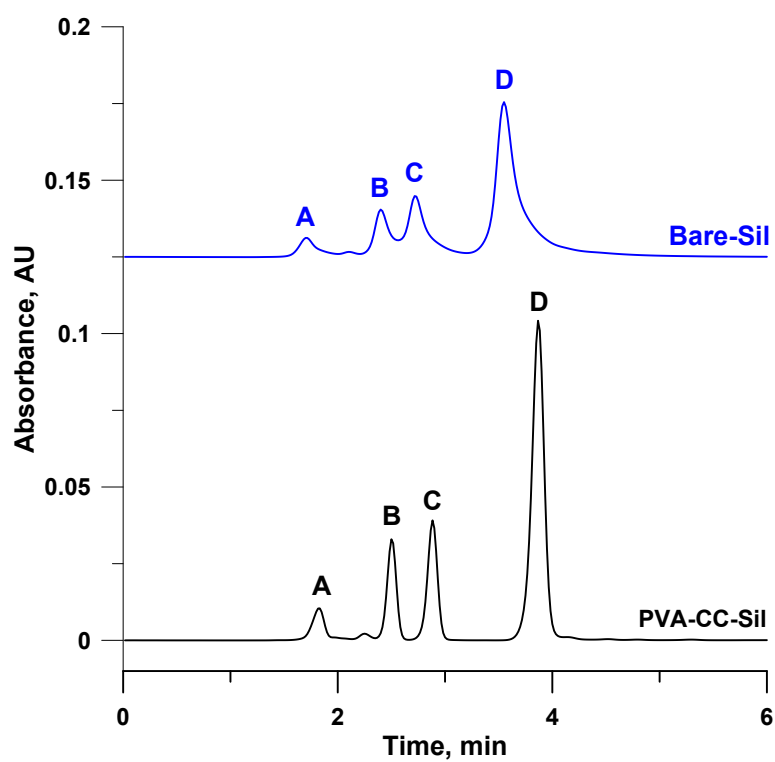
SI-Fig. 4 Comparative separation chromatograms of aromatic acids on PVA-CC-Sil, Acclaim HILIC-10, Accucore HILIC and Acchrom XCharge SAX.

Conditions: 90% ACN/10% H₂O (250 mM HCOONH₄, pH 3.48) for PVA-CC-Sil, Accucore-HILIC and Acchrom XCharge SAX; 95% ACN/5% H₂O (250 mM HCOONH₄, pH 3.48) for Acclaim HILIC-10; other conditions same as Fig. 3a.



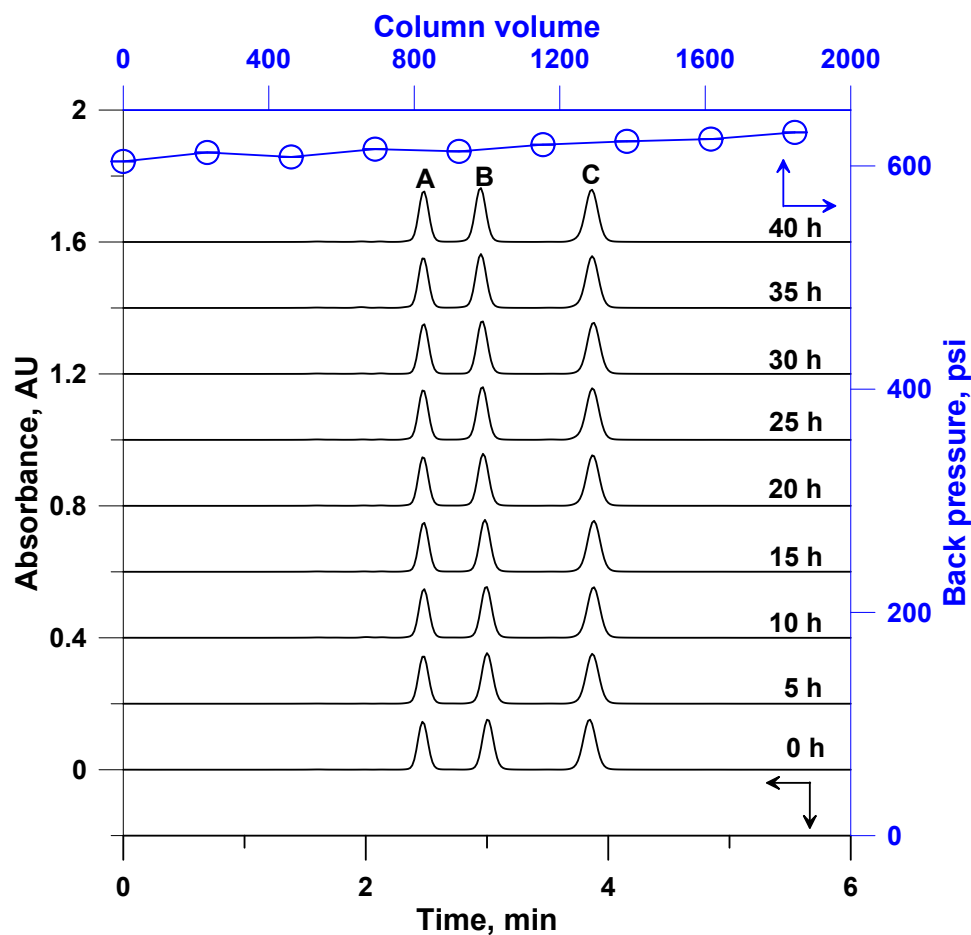
SI-Fig. 5 Comparative separation chromatograms of nucleotides on PVA-CC-Sil, Acclaim HILIC-10, Accucore HILIC and Accrom XCharge SAX.

Condition same as Fig. 4.



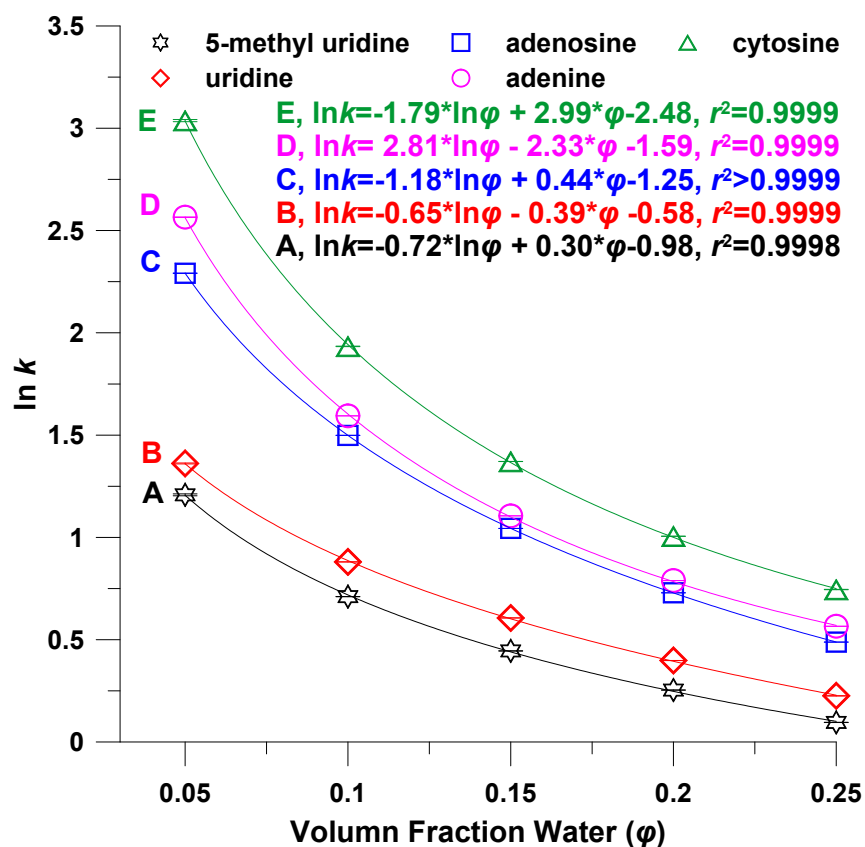
SI-Fig. 6 Comparative separation chromatograms of small molecular bases on PVA-CC-Sil and bare-sil.

Conditions: 90% ACN/10% H₂O (250 mM HCOONH₄, pH 5.87); analytes, A, 4-methyl aniline; B, caffeine; C, theobromine; D, diprophyline; injection volume, 5 μ L; other conditions same as Fig. 3.



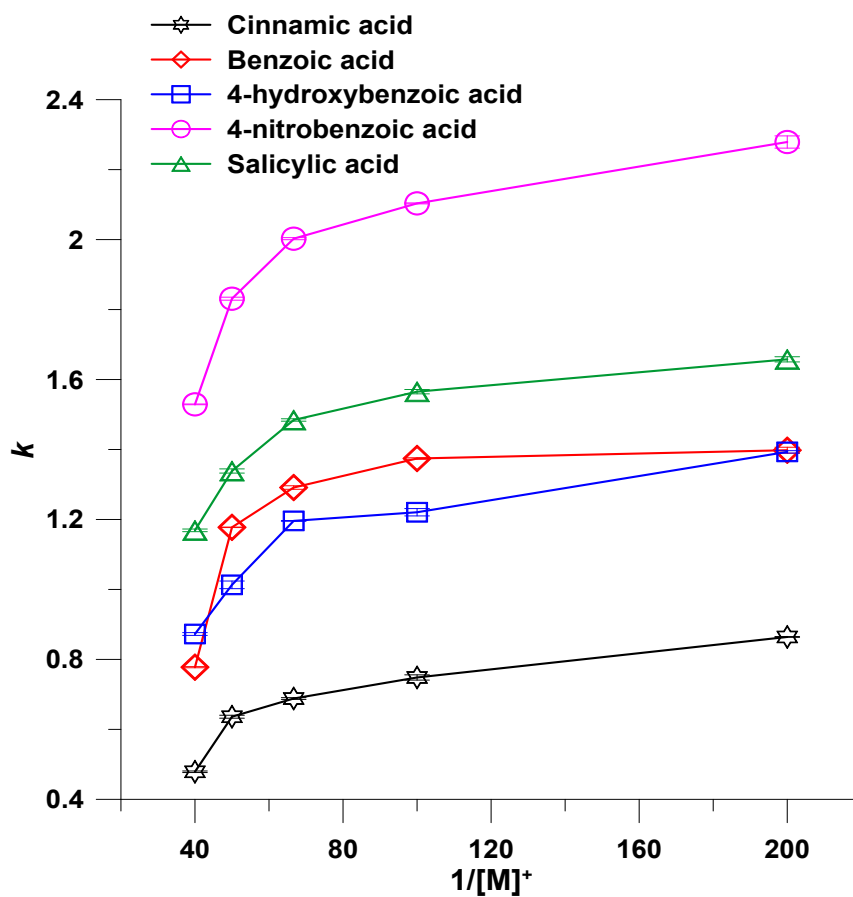
SI-Fig. 7 Chemical stability of PVA-CC-Sil stationary phase.

Conditions: mobile phase, 75% ACN/25% H₂O (20 mM NH₄OAc, pH 6.87); analytes, A, diprophyline; B, 4-aminobenzoic acid; C, cytidine; injection volume, 5 μ L; other conditions same as Fig. 3.



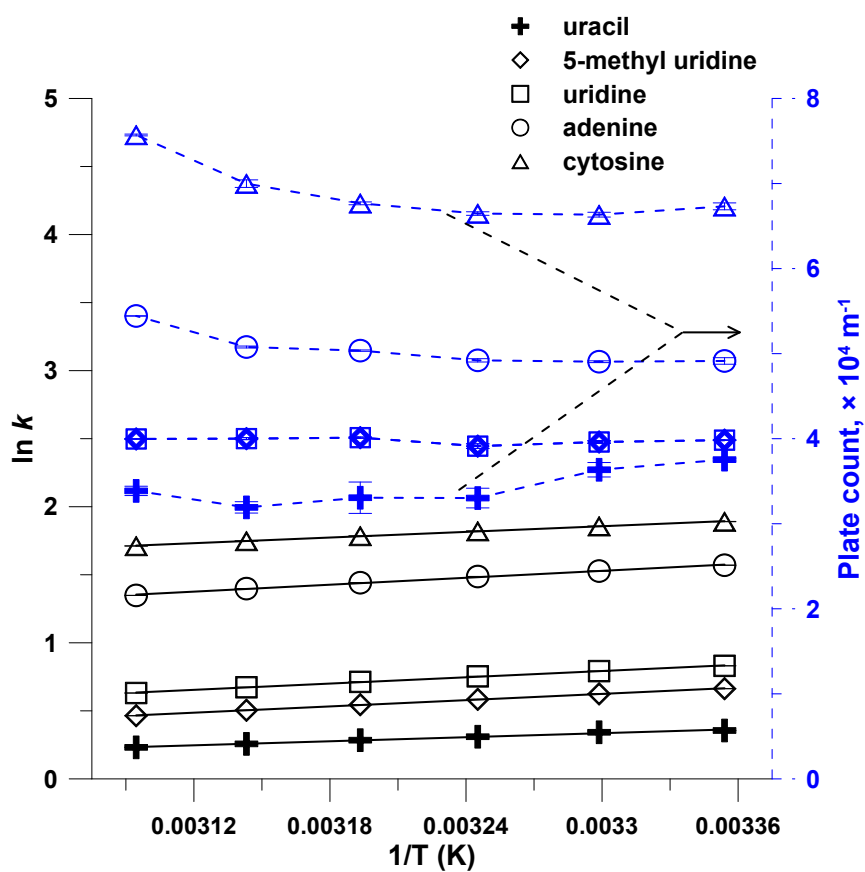
SI-Fig. 8 Plots of $\ln k$ vs. volume fraction water in mobile phase for nucleosides onto PVA-CC-Sil.

Conditions: mobile phase of ACN/H₂O varied as indicated; other conditions same as Fig. 3.



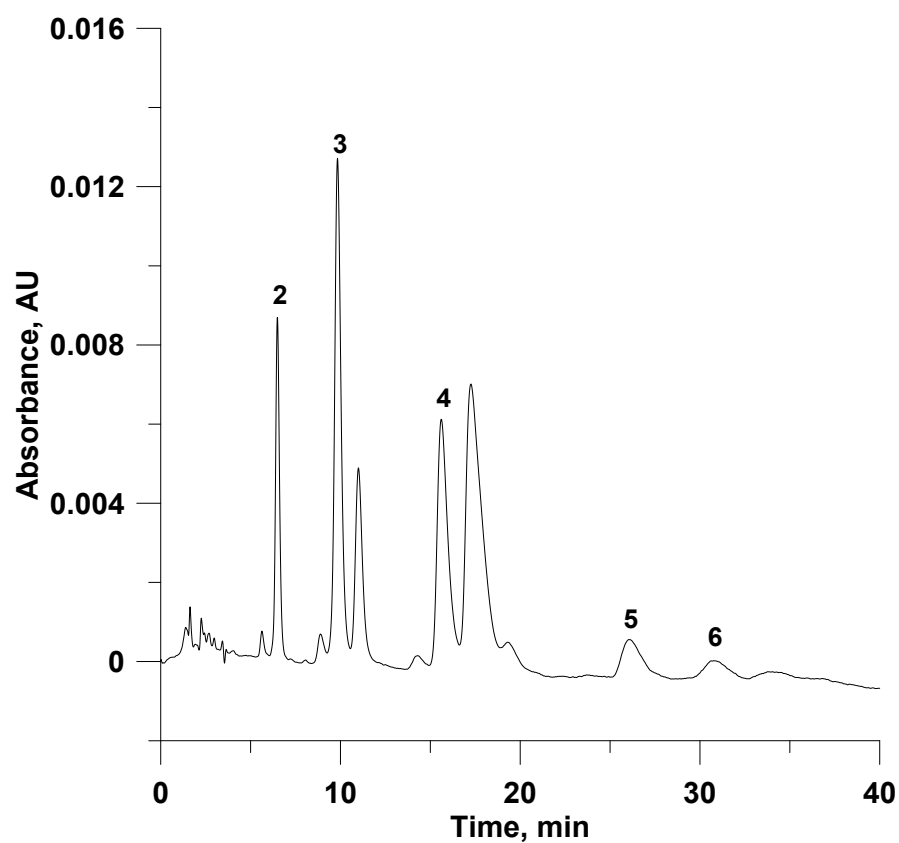
SI-Fig. 9 Plots of retention factors vs. the reciprocal of eluent electrolyte concentration on the retention for five aromatic acids onto PVA-CC-Sil.

Conditions: mobile phase, HCOONH_4 , concentration as noted, pH 3.48 with 90% ACN; other conditions same as Fig. 3.



SI-Fig. 10 Retention factor dependency on column temperature for five nucleosides and nucleic acid bases onto PVA-CC-Sil.

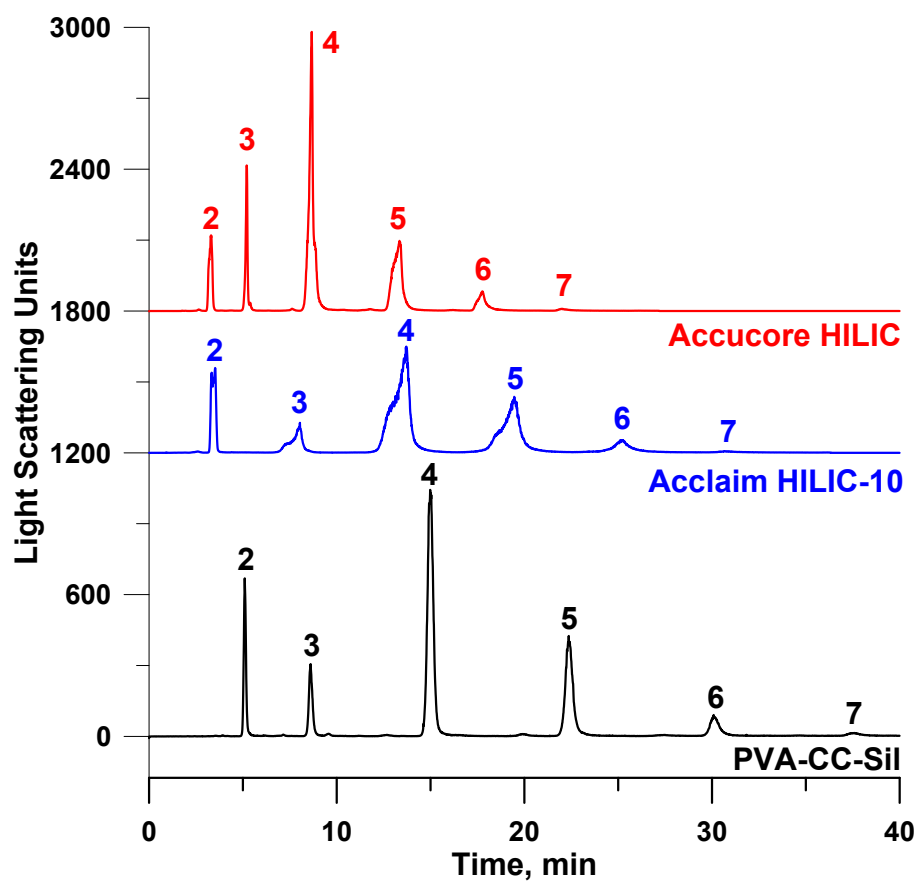
Conditions: mobile phase, 90% ACN/ 10% H₂O; temperature as indicated; other conditions same as Fig. 3.



SI-Fig. 11 Chromatogram of sodium alginate onto PVA-CC-Sil.

Condition: mobile phase, A, H₂O; B, ACN; C, 250 mM HCOONH₄, A/B/C (v/v/v), 15/65/20;

injection volume, 5 μ L; other conditions same as Fig. 3.



SI-Fig. 12 Comparative separation chromatograms of galacto-oligosaccharides on PVA-CC-Sil, Acclaim HILIC-10 and Accucore HILIC.

Conditions: mobile phase, A, 100 mM HCOONH₄; B, ACN; C, MeOH, 0-60 min, A/B/C (v/v/v), 10/70/20→10/30/60 for PVA-CC-Sil, 10/90/0→10/50/40 for Acclaim HILIC-10 and 10/80/10→10/40/50 for Accucore HILIC; other conditions same as Fig. 6.