Kromasil® AmyCoat™ and CelluCoat™ Chiral Phases

- 3, 5 and 10µm particle sizes
- Negligible memory effects
- Fully scaleable
- Suitable for HPLC and SFC applications

Kromasil® AmyCoat™ and CelluCoat™ are new generation polysaccharide-based chiral phases. High purity deactivated wide pore silica is coated with tris-(3,5-dimethylphenyl)carbamoyl functionalised amylose or cellulose selector (see Figure 10). This is performed using a unique deposition procedure.

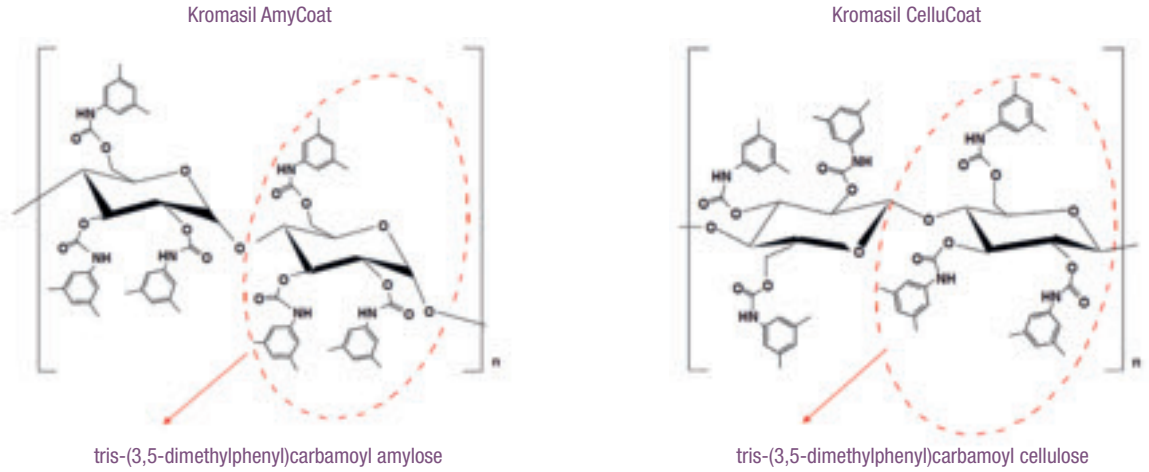


Figure 10. Structures of Kromasil AmyCoat and CelluCoat

Features of Kromasil AmyCoat and CelluCoat

1) Excellent resolution and selectivity – Both Kromasil AmyCoat and Kromasil CelluCoat show excellent enantioselectivity for many different racemates. The availability of 3µm particles enables higher efficiency and resolution for analytical separations. This improved resolution allows a higher flow rate for faster analyses. The example in Figure 11 shows baseline separation of the enantiomers of Tröger's base in less than 1 minute.

2) Fully back-integrated – Akzo Nobel manufacture the super wide pore silica for Kromasil AmyCoat and Kromasil CelluCoat and perform all subsequent steps leading to the final product. This means that the products have full traceability and the complete process is controlled.

3) Increased pressure limits – Kromasil AmyCoat and CelluCoat phases can withstand flow rates equivalent to pressures up to 400bar – the approximate upper limit for most HPLC systems. This enables faster analyses to be performed.

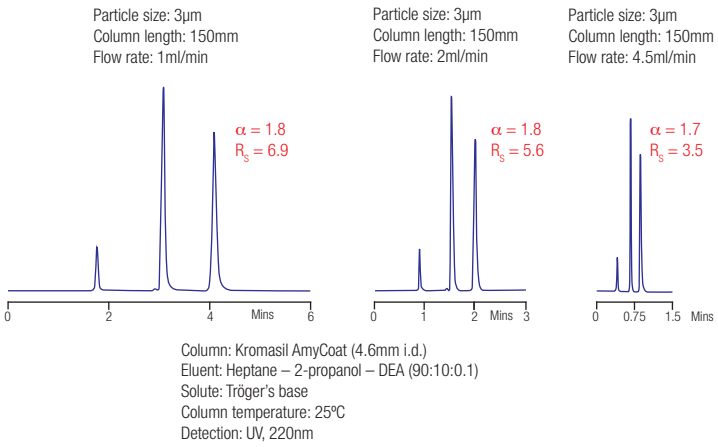
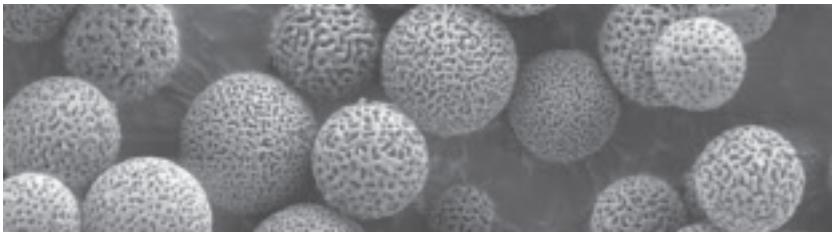


Figure 11. Fast analytical chromatography on Kromasil AmyCoat

4) Stable performance – Switching between compatible normal-phase and polar organic eluents will not lead to any degradation in the performance of these phases. This means that columns do not need to be dedicated to a particular solvent.

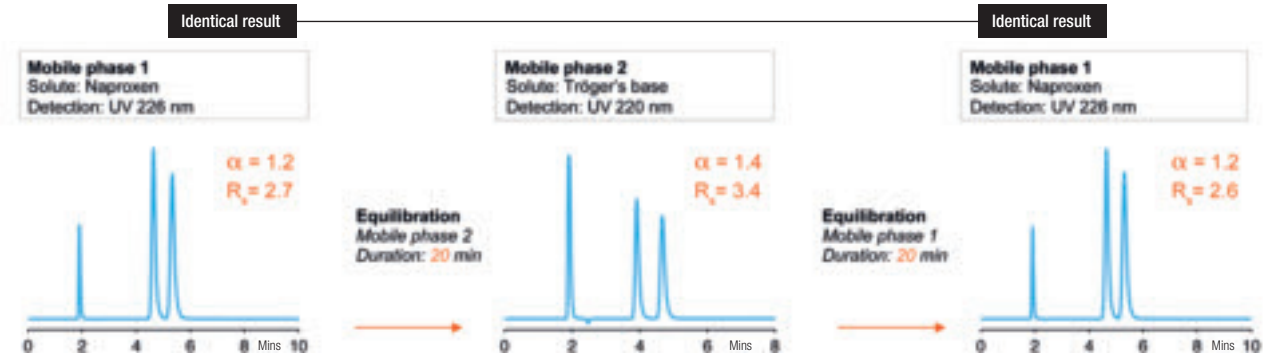
5) RP Phases – The newer Kromasil CelluCoat RP and AmyCoat RP phases are based on the same silica matrix and chiral selector as the corresponding Kromasil CelluCoat and AmyCoat phases. These 3µm particle size materials are designed for fast, efficient and high resolution analyses under reversed-phase conditions.



Kromasil® AmyCoat™ and CelluCoat™ Chiral Phases (continued)

6) Short equilibration times

The ability to use higher flow rates leads to short equilibration times. In general, long equilibration times are most pronounced when switching eluents containing basic additives to acidic additives and vice versa. Figure 12 illustrates the short equilibration times required for Kromasil CelluCoat when switching additives.



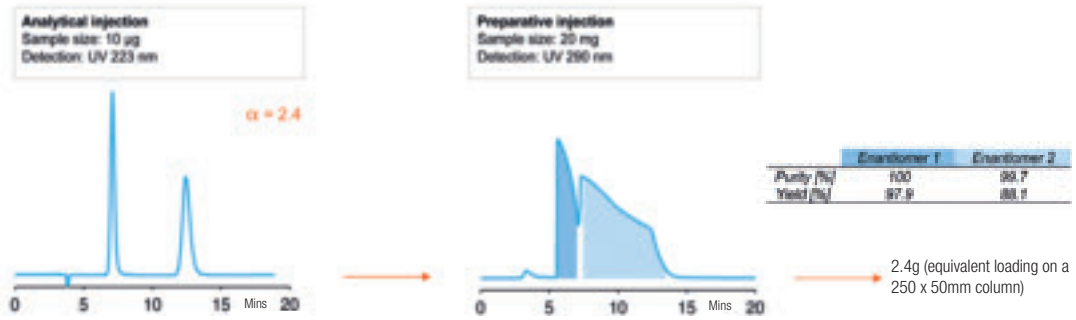
Column: Kromasil CelluCoat (3µm, 150 x 4.6mm)
Eluent 1: Heptane – 2-propanol – TFA (90:10:0.1)
Eluent 2: Heptane – 2-propanol – DEA (90:10:0.1)
Flow rate: 1ml/min
Column temperature: 25ºC

Figure 12. Short equilibration times

7) Easy to scale-up

With particle sizes from 3 to 10µm giving identical selectivity, Kromasil AmyCoat and Kromasil CelluCoat make it easy to scale-up whilst retaining excellent performance. Method development can be performed using 3µm analytical scale columns and translating the generated data to a larger column packed with 10µm particles. However, if it is known from the outset that you plan to scale-up the process, a 10µm analytical column would be recommended for method development.

Figure 13 illustrates the preparative scale-up for the analysis of metoprolol on Kromasil CelluCoat. High yields and enantiomeric purity were achieved on a 250 x 4.6mm column. If this separation were to be scaled up to a 250 x 50mm i.d. column, the equivalent loading of metoprolol would be 2.4g.



Column: Kromasil CelluCoat (10µm, 250 x 4.6mm)
Eluent: Heptane – 2-propanol – DEA (90:10:0.1)
Flow rate: 1ml/min
Column temperature: 25ºC
Solute: Metoprolol

Figure 13. Scale-up on Kromasil CelluCoat

Ordering Information

Kromasil Phase	Column Dimensions[1] (mm)				
	50 x 4.6	150 x 4.6	250 x 4.6	250 x 10.0	250 x 21.2
3µm					
AmyCoat	C03ACA05	C03ACA15	-	-	-
CelluCoat	C03CCA05	C03CCA15	-	-	-
AmyCoat RP	C03ARA05	C03ARA15	-	-	-
CelluCoat RP	C03CRA05	C03CRA15	-	-	-
5µm					
AmyCoat	C05ACA05	C05ACA15	C05ACA25	C05ACP25	C05ACQ25
CelluCoat	C05CCA05	C05CCA15	C05CCA25	C05CCP25	C05CCQ25
10µm[2]					
AmyCoat	C10ACA05	C10ACA15	C10ACA25	C10ACP25	C10ACQ25
CelluCoat	C10CCA05	C10CCA15	C10CCA25	C10CCP25	C10CCQ25

[1] Other column dimensions available on request
[2] 10µm bulk material also available

Please contact Hichrom for availability of guard cartridges.

Kromasil® DMB and TBB Chiral Phases

- Kromasil base silica
- High chemical stability
- High loadability
- Complementary phase selectivity

Akzo Nobel can supply the chiral phases Kromasil® DMB and Kromasil TBB, in which acylated N,N'-diallyl-L-tartardiamide network polymers have been covalently bound to Kromasil. Two key steps are involved in their synthesis. The chiral monomers are reacted with a multifunctional hydrosilane yielding a network polymer incorporating a bifunctional C2-symmetric chiral selector (Figure 14). The chiral polymer is then covalently bonded to functionalised Kromasil silica as shown in Figure 15.

Kromasil DMB and TBB Chiral Phases

	Kromasil DMB	Kromasil TBB
Chiral Monomer	O,O'-bis (3,5-dimethylbenzoyl)-N,N'-diallyl-L-tartardiamide	O,O'-bis (4-tert-butylbenzoyl)-N,N'-diallyl-L-tartardiamide
Particle Size (µm)	5, 10	5, 10
Surface Area (m^2/g)	340	340
Pore Size (Å)	100	100
Carbon Load (%)	14.5	15.0
Nitrogen Load (%)	0.6	0.6

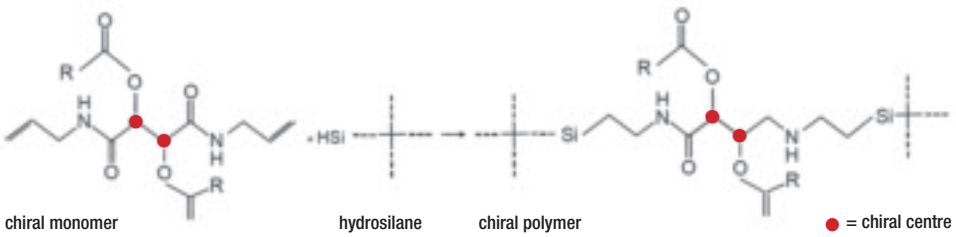


Figure 14. Chiral polymer synthesis

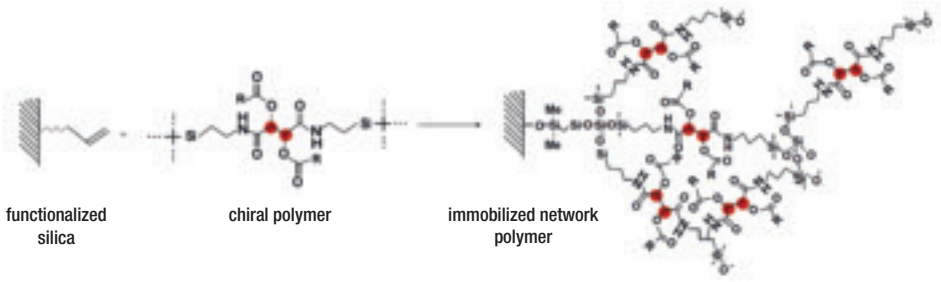


Figure 15. The binding of the chiral polymer to the Kromasil silica

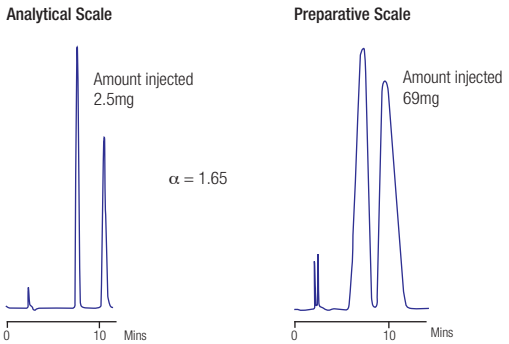
Features of Kromasil DMB and TBB

1) Mechanical and Chemical Stability

Kromasil silica is one of the most mechanically stable HPLC silicas. The high chemical stability is due to the nature of the network polymer covalently bonded to the silica. Kromasil chiral phases can be used with most solvents and buffers in the eluent without degradation.

2) Loadability

The high loading capacity of Kromasil chiral columns is due to the high surface area of the silica and the high chiral ligand density. An example of the loading capacity in preparative scale is shown for naproxen in Figure 16.



Column: Kromasil TBB (250 x 10mm)
Eluent: Hexane - acetone (92:8) + 1.0% acetic acid
Flow rate: 4.7ml/min

Figure 16. Preparative separation of (+/-)-naproxen

Kromasil® DMB and TBB Chiral Phases (continued)

3) Selectivity

Kromasil® DMB and TBB separate a broad range of racemates and have been developed to complement each other in selectivity. For the separation of acidic racemates, it is recommended to start with the TBB phase. The table below shows a series of acidic, basic and neutral racemates evaluated on both phases under identical eluent conditions.

Compound	Separation Factor (α)	
	DMB	TBB
Oxazepam	1.13	1.35
Lorazepam	1.49	1.50
Baclaphenlactam	1.32	1.53
Binaphtol	2.75	1.23
Paraflutizide	1.34	1.18
Bendroflumetiazide	1.32	1.24
Proglumide	1.26	1.56
Naproxen	1.11	1.65
Ibuprofen	1.12	1.51
Bupivacaine	1.34	1.92
Tocainide	1.12	1.33

Figures 17 and 18 show applications of Kromasil TBB and DMB chiral columns respectively.

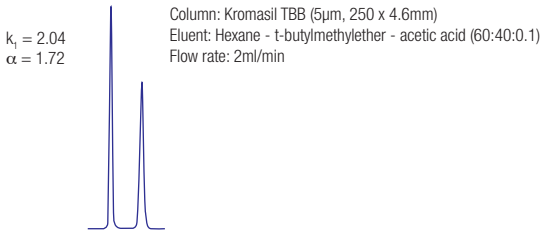


Figure 17. Analysis of benoxaprofen on Kromasil TBB

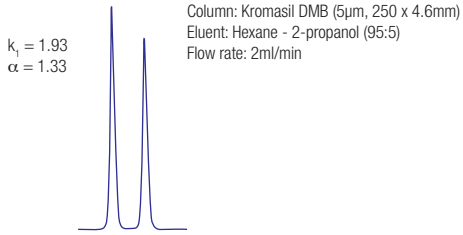


Figure 18. Analysis of mephenytoin on Kromasil DMB

Ordering information

Chiral Phase	Column Dimensions (mm)		
	250 x 4.6	250 x 10.0	250 x 21.2
5µm Phase	**POA**	**POA**	**POA**
Kromasil DMB	C05DMA25	C05DMP25	C05DMQ25
Kromasil TBB	C05TBA25	C05TBP25	C05TBQ25
10µm Phase	**POA**	**POA**	**POA**
Kromasil DMB	C10DMA25	C10DMP25	C10DMQ25
Kromasil TBB	C10TBA25	C10TBP25	C10TBQ25

Kromasil Chiral Method Development Kits are available – please enquire for details.
Please contact Hichrom for availability of guard cartridges.