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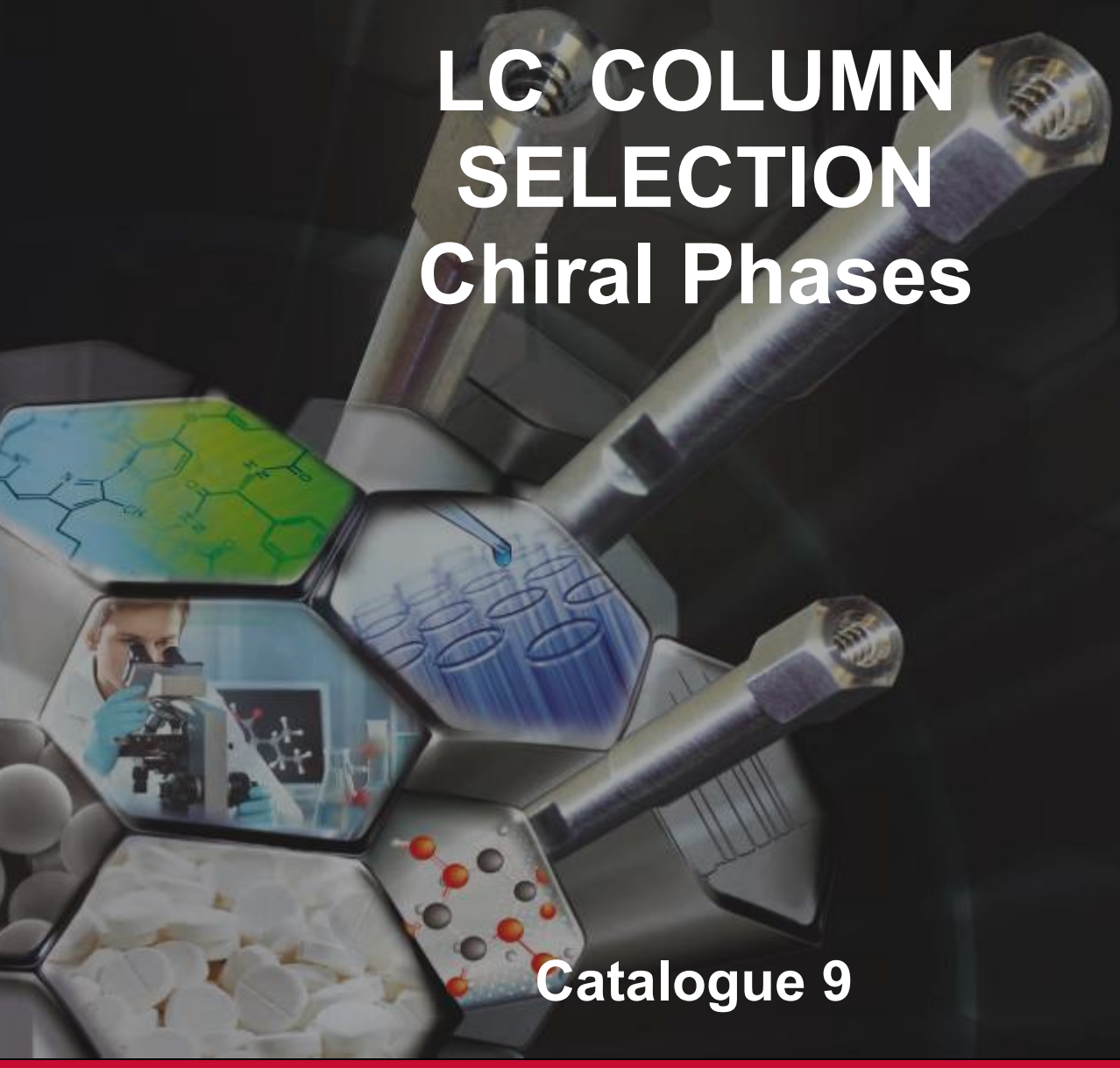
Chromatography Columns and Supplies

LC COLUMN SELECTION Chiral Phases



Catalogue 9

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CHIRAL PHASES

In many biological processes, the activity of one member of an enantiomeric pair can be contrasted with the inactivity or even harmful activity of the other. The successful development of chiral stationary phases (CSPs) for HPLC and SFC now allows us to monitor the optical purity of a bulk drug and its presence in formulations or biological fluids. Further applications can be found within the agrochemical and related industries. The main types of HPLC/SFC CSPs are discussed below, with examples listed on pages 56-57. Please see pages 286, 287 and 293 for GC chiral phases.

Immobilised Polysaccharide CSPs

Coated polysaccharide CSPs are limited in the solvents that may be used in the eluent and as sample diluents. Newer immobilised CSPs allow the use of a more robust and expanded range of solvents and bring new selectivity and higher sample solubility relative to conventionally coated CSPs.

Cellulose and Amylose Bound

Cellulose and amylose are linear polymers of optically active glucose units with molecular weights of 250,000 to 1,000,000. Cross-linked derivatives of these materials coated onto silica give unique chiral selectivity. Their chiral recognition properties depend on the 'steric fit' of guest enantiomers into the material's cavities. Choice of eluent is the key factor affecting chiral recognition.

'Brush-Type'

Although 'brush-type' (Pirkle) chiral selectors are relatively simple molecules, their well defined structure contains three types of functional groups capable of participating in charge transfer (π-π bonding), hydrogen bonding ('dipole stacking' interactions) and steric effects. The monolayer of chiral selector covalently bound to the silica surface usually gives a column of relatively high capacity and efficiency but often with limited chiral discrimination ability. Since the synthesis of the popular D-3,5-dinitrobenzoylphenylglycine phase, significant numbers of these multiple interaction CSPs have been synthesised. Polyaromatic hydrocarbon derivative CSPs are the most recent additions to the range. All 'brush-type' phases are typically used with normal-phase eluents.

Protein Bound

Proteins are high molecular weight polymers containing chiral sub-units. When bound to silica they act as very effective CSPs. The binding or complexation of small enantiomeric molecules is often stereospecific, especially for serum proteins such as α_1-acid glycoprotein (AGP) or human serum albumin (HSA). The additional stability of the Ultron ES-OVM and ES-Pepsin columns enable them to be used with high organic content eluents. Immobilised enzymes can similarly be used. Protein immobilised CSPs are typically used in buffered aqueous eluents compatible with many biological samples. They offer good selectivity. Enantiomer retention and stereoselectivity can often be significantly altered by changes in eluent pH or modifier concentration. Their low capacity makes them unsuitable for preparative applications.

Cyclodextrin Inclusion

Cyclodextrins are a class of oligosaccharides containing six to twelve optically active glucose units. They are covalently bound to silica to form the corresponding CSP. The physical shape of these molecules is that of a truncated cone, the internal diameter of which is proportional to the number of glucose units. The interior of the cavity is relatively hydrophobic. Secondary hydroxyl groups at the entrance to the cavity contribute to the separation process. The relative stability of the inclusion complexes formed by the enantiomers of the guest molecule at the edge of the cyclodextrin cavity dictates the degree of separation. β-Cyclodextrin and its derivatives are the most commonly used CSPs of this type. Cyclodextrin CSPs are used in reversed-phase and are suitable for preparative separations.

Crown Ether

Chiral recognition with crown ether phases is achieved when a complex is formed between the crown ether and an ammonium ion from the analyte. These phases are used for solutes with a primary amino group at or near its chiral centre, such as amino acids and amino alcohols.

Ligand Exchange

Ligand exchange chiral phases are characterised by the attachment of a chiral chelating ligand to the stationary support. In the presence of an appropriate transition metal cation such as copper (II), a molecular complex is formed with the chiral stationary phase ligand and the analyte. Compounds that are suitable for chiral ligand exchange are α-amino acids, hydroxy acids and small peptides.

Network Polymeric

In a network polymeric CSP the chiral selector is anchored into a network polymer by a cross-linking reaction which simultaneously bonds it to the silica. The aim is to combine in one CSP the efficiency and capacity of 'brush-type' structures with the chiral recognition power of those phases based on chiral polymers.

Chiral Phases (continued)

Chiral Phases

Phase	Manufacturer	Chiral Type	Chiral Selector	Particle Size (µm)	Features	Page
CHIRA-chrom-1	Hichrom	Brush	D-Phenylglycine	5	High efficiency and capacity. Low cost	140
			L-Phenylglycine	5		140
			L-Leucine	5		140
CHIRA-chrom-2			Dinitrophenyltartramide	5		140
ChiraDex	Merck	Cyclodextrin	β-Cyclodextrin	5	Forms inclusion complexes	180
CHIRALPAK AGP	Chiral Technologies[2]	Protein	α_1-Acid glycoprotein	5	Widely used. pH variation a useful tool	88, 89
CHIRALPAK CBH		Enzyme	Cellobiohydrolase	5		88, 89
CHIRALPAK HSA		Protein	Human serum albumin	5		88, 89
CHIRALPAK IA		Amylose	Immobilised amylose derivative	3, 5	Broad application range	81, 82, 86, 87
CHIRALPAK IB		Cellulose	Immobilised cellulose derivative	3, 5		81, 82, 86, 87
CHIRALPAK IC		Cellulose		3, 5		81, 82, 86, 87
CHIRALPAK ID		Amylose	Immobilised amylose derivative	3, 5		81, 82, 86, 87
CHIRALPAK IE		Amylose		3, 5		81, 82, 86, 87
CHIRALPAK IF		Amylose		3, 5		81, 82, 86, 87
CHIRALPAK AD		Amylose	Amylose derivative	3, 5, 10	Unique separation applications. Very versatile	83, 84, 86, 87
CHIRALPAK AS				3, 5, 10		83, 84, 86, 87
CHIRALCEL OD		Cellulose	Cellulose derivative	3, 5, 10		83, 84, 86, 87
CHIRALCEL OJ				3, 5, 10		83, 84, 86, 87
CHIRALPAK QD-AX		Anion-exchange	Quinidine derivative	5	Useful for chiral acids	85
CHIRALPAK QN-AX			Quinine derivative	5		85
CROWNPAK		Crown ether	18-crown-6 type crown ether	5	Suitable for amino acids and primary amines	90
Chirobiotic R	Supelco[1]	Macrocyclic glycopeptide	Ristocetin A	5	Broad selectivity	-
Chirobiotic T			Teicoplanin	5		-
Chirobiotic V			Vancomycin	5		-
Cyclobond I		Cyclodextrin	β-Cyclodextrin	5	Forms inclusion complexes	-
Cyclobond II			γ-Cyclodextrin	5		-
ChiroSil	Regis/RStech	Crown ether	(18-crown-6)-tetracarboxylic acid	5, 10	Suitable for primary amines and amino acids	208
DACH-DNB	Regis	Brush	3,5-Dinitrobenzoyl derivatives	5	π-electron acceptor/donor. Widely used	204, 205, 207
ULMO				5		204, 205, 207
Whelk-01/Whelk-02				5, 10		204, 205, 207
α-Burke 2				5	π-electron acceptor	204, 207
β-GEM 1				5		204, 207
Leucine				5		204, 207
Phenylglycine				5		204, 207
Pirkle-1J			β-Lactam derivative	5		204, 207
RegisCell		Cellulose	Cellulose derivative	5, 10	Broad application range	206, 207
RegisPack		Amylose	Amylose derivative	5, 10		206, 207
RegisPack CLA-1		Amylose	Chlorinated amylose derivative	10	Complementary selectivity to RegisCell and RegisPack	206, 207
Kromasil AmyCoat	Akzo Nobel	Amylose	Amylose derivative	3, 5, 10	Broad application range	152, 153
Kromasil CelluCoat		Cellulose	Cellulose derivative			152, 153
Kromasil DMB		Network polymer	Acylated N,N'-diallyl-L-tartardiamide	5, 10	High stability and capacity. Suitable for preparative applications	154, 155
Kromasil TBB				5, 10		154, 155

[1] Please contact Hichrom for ordering information

[2] CHIRALPAK ZWIX phases also available – see page 5

Chiral Phases (continued)

Chiral Phases (continued)

Phase	Manufacturer	Chiral Type	Chiral Selector	Particle Size (µm)	Features	Page
NUCLEODEX β-OH	Macherey-Nagel	Cyclodextrin	β-Cyclodextrin	5	Reversed-phase applications	169
NUCLEODEX α-PM			Permethylated α-, β- and γ-cyclodextrins respectively	5		169
NUCLEODEX β-PM				5		169
NUCLEODEX γ-PM				5		169
NUCLEOSIL CHIRAL-1		Ligand exchange	L-Hydroxyproline-Cu^{2+} complex	5	α-Amino acid applications	169
RESOLVOSIL BSA-7		Protein	Bovine serum albumin	7		169
NUCLEOCEL *DELTA*		Cellulose	Cellulose derivative	5	Broad application range	169, 170
ORpak CDA	Shodex	Cyclodextrin	α-Cyclodextrin	6	Polyhydroxymethacrylate base material	214
ORpak CDB			β-Cyclodextrin	6		214
ORpak CDC			γ-Cyclodextrin	6		214
ORpak CDBS			β-Cyclodextrin	3	Silica base	214
ORpak CRX		Ligand exchange	L-Amino acid derivative	6	Suitable for underivatised amino acids	214
Ultron ES-OVM	Shinwa Chemical Industries	Protein	Ovomucoid	5, 10	USP L57 column	260
Ultron ES-Pepsin			Pepsin	5	Suitable for basic compounds	260
Ultron ES-CD		Cyclodextrin	β-Cyclodextrin	5	Suitable for hydrophobic cyclic compounds	260
Ultron ES-PhCD			Phenylcarbamated β-cyclodextrin	5		260
YMC Chiral CD BR	YMC[3]	Cyclodextrin	Bromide derivatives of cyclodextrin (α, β or γ)	5	Separates wide range of polar compounds	274
YMC Chiral NEA		Brush	α-Naphthylethylamine	5	NP or RP applications	274
YMC Sumichiral OA series		Various	Various	5	17 different phases	274
ZirChrom Chiral LEU	ZirChrom	Brush	Leucine derivative	3, 5	Zirconia base material	277
ZirChrom Chiral NESA			Naphthylethylsuccinamic acid derivative	3, 5		277
ZirChrom Chiral PG			Phenylglycine derivative	3, 5		277
ZirChrom CelluloZe		Cellulose	Cellulose derivative	3, 5		277

[3] YMC CHIRAL polysaccharide phases also available – see page 5