HICHROM

Chromatography Columns and Supplies

LC COLUMNS
Regis
Chiral Phases

Catalogue 9

Hichrom Limited

1 The Markham Centre, Station Road
Theale, Reading, Berks, RG7 4PE, UK

Tel: +44 (0)118 930 3660 Fax: +44 (0)118 932 3484

Email: sales@hichrom.co.uk www.hichrom.co.uk

REGIS® TECHNOLOGIES

- Wide range of brush-type chiral phases
- Polysaccharide based phases
- Free chiral screening service
- Chiral application guide
- IAM and RAM phases
- GC derivatisation reagents
- Ion pairing reagents

Regis® Technologies manufactures a wide range of chiral and speciality HPLC and SFC columns for analytical to preparative applications and supplies the ChiroSil® range of crown ether type chiral columns manufactured by RStech Corporation, Korea (see page 208). RegisCell®, RegisPack® and RegisPack CLA-1™ are polysaccharide phases, described on page 206.

Pirkle Chiral Phases

- Column durability due to covalent bonding
- Ability to invert elution order
- Excellent chromatographic efficiency
- Universal solvent compatibility
- Analytical to preparative dimensions
- Enantiomer separation of wide variety of compound types
- Applicable for HPLC and SFC methods

Regis Technologies is the leading manufacturer of 'brush-type' or Pirkle concept chiral stationary phases, for use in both normal-phase and reversed-phase modes. For each phase, the optically active ligand is covalently bonded to 5µm, 100Å spherical silica.

Regis Pirkle Chiral Phases

Phase	Bonding	Class	Typical Applications
α-Burke 2	N-3,5-dinitrobenzoyl-α-amino-2,2-dimethyl-4-pentenyl phosphonate	π-electron acceptor	β-Blockers, amino alcohols
β-Gem 1	N-3,5-dinitrobenzoyl-3-amino-3-phenyl-2-(1,1-dimethylethyl)-propanoate	π-electron acceptor	Anilide derivatives of wide range of carboxylic acids
DACH-DNB	3,5-Dinitrobenzoyl derivative of 1,2-diaminocyclohexane	π-electron acceptor/donor	Broad range
Leucine	3,5-Dinitrobenzoylleucine	π-electron acceptor	Benzodiazepines
Phenylglycine	3,5-Dinitrobenzoylphenylglycine	π-electron acceptor	Wide variety of compounds containing π-basic groups
Pirkle 1-J	3-(3,5-Dinitrobenzamido)-4-phenyl-β-lactam	π-electron acceptor	Underivatised β-blockers, arylpropionic acids
ULMO	3,5-Dinitrobenzoyl derivative of diphenylethylene-diamine	π-electron acceptor/donor	Wide range, particularly aryl carbinols
Whelk-01 Whelk-02	1-(3,5-dinitrobenzamido)-tetrahydrophenanthrene	π-electron acceptor/donor	Broad range of compounds

Features of Pirkle Phases

1) Inversion of elution order

An important advantage of the Pirkle chiral stationary phases (CSP) is the ability to invert elution order by using the same type of CSP, but with the opposite absolute configuration. As a result, by selecting the phase with the optimum configuration, it is possible to have the trace enantiomer elute before the major, which is beneficial for enantiomeric purity determinations and for preparative separations.

2) Analytical and preparative columns

All of Regis' Pirkle columns are available in both analytical and preparative sizes. The high loading factors offered by these phases make them particularly suited for scaling up.

3) Enantiomeric purity determination

Pirkle CSPs are particularly useful for the accurate determination of enantiomeric purity, especially in trace analysis because they provide excellent resolution. Such determinations are fast, accurate and sensitive.

4) Universal solvent compatibility

Pirkle HPLC columns are compatible with most HPLC eluents with pH between 2.5 and 7.5. Although superior resolution is typically observed with normal-phase elution, many racemates can be separated in either normal- or reversed-phase mode. The separation of enantiomers using SFC with Pirkle columns is now well proven.

Pirkle Chiral Phases (continued)

Whelk-0®1 and Whelk-0®2

Whelk-0®1 is the most widely applicable chiral phase, due to the incorporation of both π-acceptor and π-donor characteristics. It was originally designed for the separation of underivatized non-steroidal anti-inflammatory drugs, but shows versatility in the analysis of a wide range of compounds, including amides, epoxides, esters, ureas, carbamates, ethers, aldehydes, ketones, carboxylic acids and alcohols.

The newer Whelk-01 phase, based on 5µm Kromasil silica, shows higher efficiencies and greater resolving power in both HPLC and SFC modes than the original 5µm Whelk-01 phase. Although the original material is still offered for the continuation of validated methods, for all other methods it is recommended that the newer Whelk-01 product is used. Figures 1 and 2 show comparisons of the HPLC and SFC performances of these Whelk-01 phases for modafinil and chlormezanone respectively.

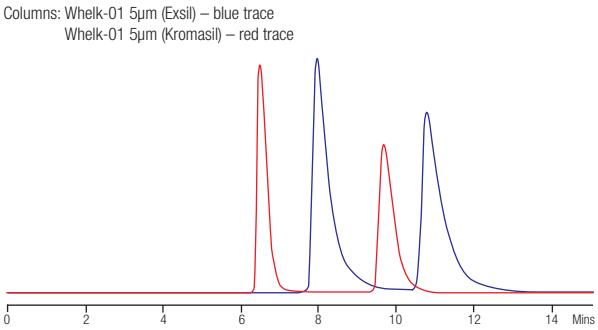


Figure 1. HPLC separation of modafinil

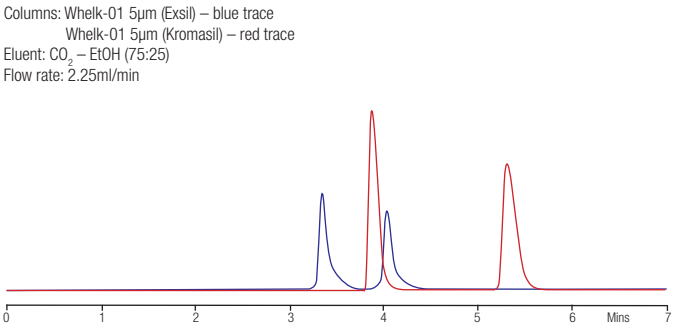
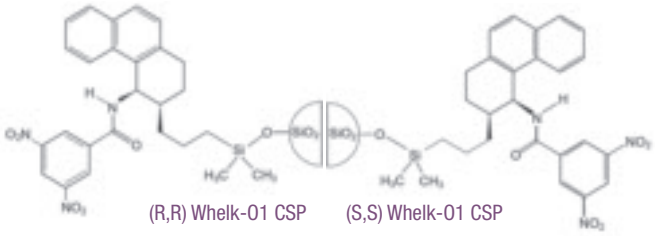
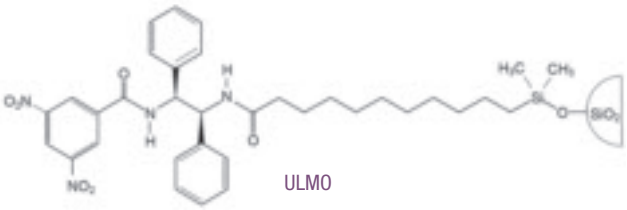


Figure 2. SFC separation of chlormezanone

Whelk-0®2 is the covalent trifunctional version of the Whelk-01. It shows similar enantioselectivity as the Whelk-01, but enhanced stability with strong organic modifiers.



(R,R) Whelk-01 CSP (S,S) Whelk-01 CSP



ULMO

ULMO

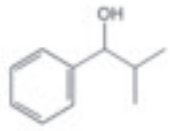
The ULMO chiral stationary phase has the general capability of separating enantiomers of many racemate classes and is particularly good at separating enantiomers of aryl carbinols.

DACH-DNB

DACH-DNB contains the 3,5-dinitrobenzoyl derivative of trans 1,2-diaminocyclohexane. It can resolve a wide range of compound classes, including sulphoxides, phosphine oxides, selenoxides, organometallics and atropisomers.

Column: (S,S)-ULMO
(250 x 4.6mm)
Eluent: Heptane - IPA (99:1)
Flow rate: 1ml/min

Run time: 6 mins
k_1: 0.86
α: 1.38



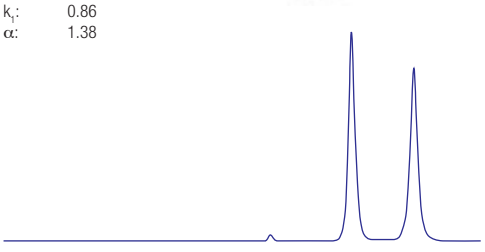


Figure 3. Separation on (S,S)-ULMO

Column: (S,S)-DACH-DNB
(250 x 4.6mm)
Eluent: CH_2Cl_2 - IPA (98:2)
Flow rate: 1ml/min

Run time: 17 mins
k_1: 3.33
α: 1.63

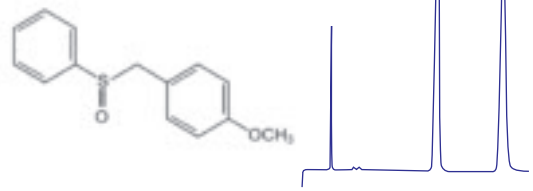


Figure 4. Separation on (S,S)-DACH-DNB

RegisCell®, RegisPack® and RegisPack CLA-1™

- Polysaccharide coated phases
- Excellent selectivity
- Fast equilibration times
- Applicable for HPLC and SFC methods

RegisCell® and RegisPack® chiral columns are polysaccharide based stationary phases. They are produced using a unique manufacturing process involving the coating of the proven chiral selectors tris-(3,5-dimethylphenyl) carbamoyl cellulose and tris-(3,5-dimethylphenyl) carbamoyl amylose respectively onto a high purity wide pore (1000Å) silica.

Columns are packed and tested at high pressures for use in both SFC and HPLC modes. The packing materials have a high pressure limit (450 bar), which enables faster flow rates and decreased equilibration times when switching from one compatible eluent to another.

RegisCell and RegisPack produce excellent performance and resolution in the separation of a wide range of compounds, by both HPLC and SFC. Figure 5 shows the separation of metalaxyl on RegisCell and Figure 6 shows the separation of methaqualone on RegisPack. Fast SFC analyses have been developed for a number of applications, using simple eluent conditions, such as shown in Figure 7 for the analysis of atenolol in less than one minute.

The newer RegisPack CLA-1, a coated chlorinated phase with the chiral selector tris-(5-chloro-2-methylphenyl) carbamoyl amylose, shows complementary selectivity to the other Regis phases. In cases where the proven chiral phases Whelk-01, RegisPack and RegisCell give incomplete separation, RegisPack CLA-1 can be a useful addition to the screening process. Figure 8 shows the separation of the four diastereomers of cyclandelate by HPLC on a RegisPack CLA-1 column.

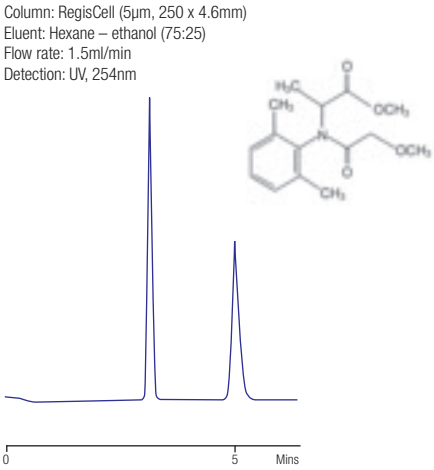


Figure 5. Separation of metalaxyl on RegisCell

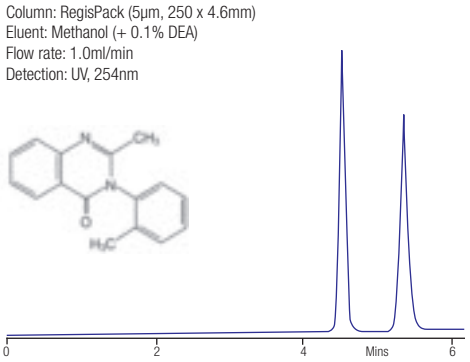


Figure 6. Separation of methaqualone on RegisPack

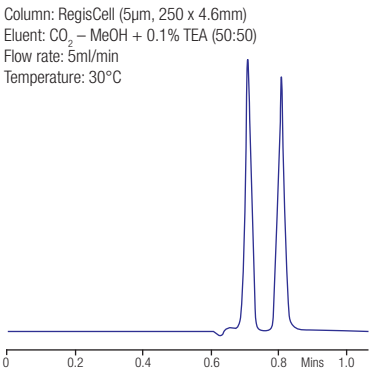


Figure 7. Fast SFC separation of atenolol

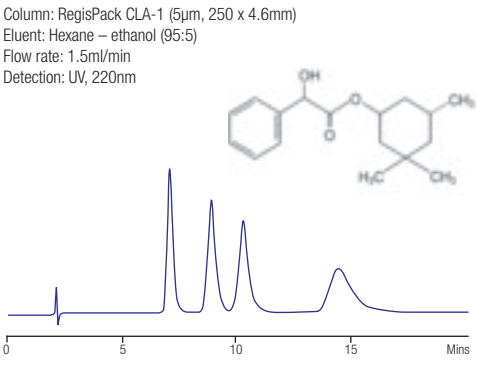


Figure 8. Cyclandelate on RegisPack CLA-1

Ordering Information – Regis Chiral Columns

Regis Phase (5µm)[1]	Column Dimensions[2] (mm)			
	50 x 2.1	50 x 4.6	250 x 4.6	250 x 10
(R,R)-α-Burke 2	731602	731613	735035	735235
(S,S)-α-Burke 2	731603	731614	735037	735237
(R,R)-β-Gem 1	731600	731611	731043	731243
(S,S)-β-Gem 1	731601	731612	731029	731229
(R,R)-DACH-DNB	788246	788248	788101	788102
(S,S)-DACH-DNB	788247	788249	788201	788202
D-Leucine	731607	731618	731054	731254
L-Leucine	731608	731619	731041	731241
D-Phenylglycine	731609	731620	731021	731221
L-Phenylglycine	731610	731621	731024	731224
(3R,4S)-Pirkle 1-J	731604	731615	731044	731244
(3S,4R)-Pirkle 1-J	731605	731616	731045	731245
(R,R)-ULMO	787648	787650	787200	787201
(S,S)-ULMO	787647	787649	787100	787101
(R,R)-Whelk-01[3]	780255	780252	780201	780202
(S,S)-Whelk-01[3]	780155	780152	780101	780102
(R,R)-Whelk-01[4]	786901	786905	786201	786202
(S,S)-Whelk-01[4]	786900	786904	786101	786102
(R,R)-Whelk-01[3] (10µm)	786903	786907	786515	786525
(S,S)-Whelk-01[3] (10µm)	786902	786906	786615	786625
(R,R)-Whelk-02[3] (10µm)	-	-	786315	786325
(S,S)-Whelk-02[3] (10µm)	-	-	786415	786425
RegisCell	-	784101	784104	784105
RegisPack	-	783101	783104	783105
RegisPack CLA-1	-	793101	793104	793105
RegisCell (10µm)	-	-	784204	784205
RegisPack (10µm)	-	783201	783204	783205
RegisPack CLA-1 (10µm)	-	793201	793204	793205

[1] Bulk material and preparative columns available [2] Other dimension columns available [3] Based on Kromasil silica [4] Based on original Exsil silica

Free Chiral Screening Service

Regis offer a free chiral screening service, where a small amount (20mg) of customer supplied sample is screened on a range of the key Regis columns. If you are interested in this service, please contact Hichrom to obtain a submission form and Confidentiality Agreement. Screening in both HPLC and SFC modes is available. Advice can also be given on scaling up chiral separations to preparative dimensions.

Ion Pairing Reagents

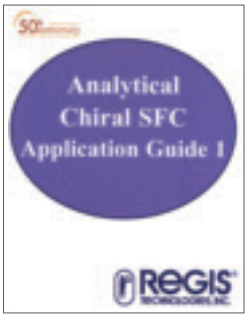
Ion pairing reagents can be used to selectively increase the retention of charged analytes on conventional hydrophobic HPLC phases. Regis manufactures both anionic sulphonate (S-Series) and cationic quaternary amine (Q-Series) ion pairing reagents with varying alkyl chain lengths. The S-Series reagents are available as 0.5M solutions of alkyl sulphonates (S5-S8, S12) or as bulk powder. The Q-Series consists of 0.5M solutions of quaternary alkyltriethylamines (Q5-Q8, Q12). Method development kits are also available.

GC Derivatization Reagents

Regis manufactures a wide range of high purity derivatization reagents for GC. These include reagents for silylation, alkylation and acylation reactions. Please see page 294 for further details and ordering information.

Regis Chiral Application Guides

A wide range of examples of enantiomer separations can be found in the Regis Chiral HPLC Application Guide and Chiral SFC Application Guide. Please contact us for a free copy.



CHIROSIL®

- Efficient analysis of amino acids and primary amines
- Excellent durability due to covalent bonding
- Ability to invert elution order
- Robust crown ether phase for HPLC

The ChiroSil® RCA(+) and SCA(-) crown ether chiral stationary phases were developed by RStech Corporation in South Korea. These specialised phases are effective in separating various natural and unnatural amino acids, as well as compounds containing a primary amino group near the chiral centre. They are also successfully used for chiral resolution of chiral amino alcohols including therapeutically active compounds such as amphetamine, phenylethanolamine, octopamine and norepinephrine.

These phases are prepared by a covalent trifunctional bonding (+) or (-)-(18-crown-6)-tetracarboxylic acid chiral selector to aminopropyl silica (see Figure 9). This results in a material which shows excellent durability and reproducibility. The high resolution capability of the phases enables applications to be scaled up from analytical to preparative dimensions. The availability of both enantiomeric forms of the phase enables the elution order to be inverted so that a trace enantiomer can be eluted first.

ChiroSil RCA(+) and SCA(-) phases

Particle Size (µm)	5, 10
Pore Size (Å)	100
Maximum Recommended Operating Temperature (°C)	50
Recommended pH Range	2 – 7.5

Figures 10 and 11 show the enantiomeric separation of glutamic acid and 1,2,3,4-tetrahydro-1-naphthylamine respectively using ChiroSil SCA(-).

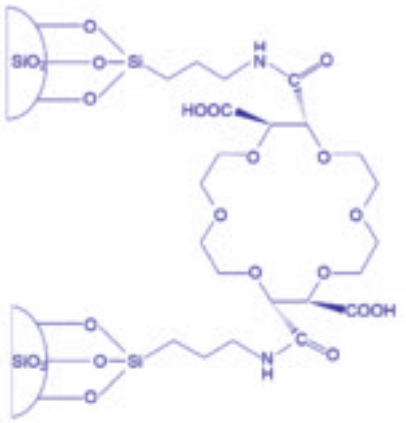
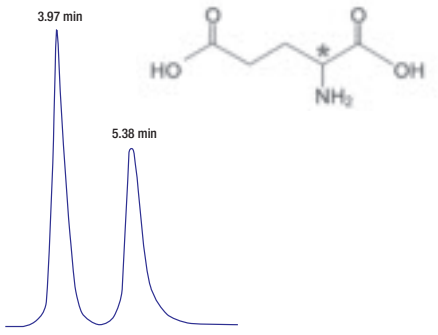
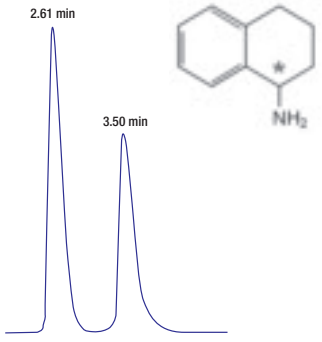


Figure 9. Bonding of ChiroSil phase



Eluent: CH_3OH - H_2O (84:16) in 5mM $HClO_4$
Flow rate: 0.8ml/min

Figure 10. Analysis of glutamic acid



Eluent: CH_3OH - H_2O (84:16) in 10mM H_2SO_4 + 1ml Et_3N
Flow rate: 1ml/min

Figure 11. Analysis of 1,2,3,4-tetrahydro-1-naphthylamine

Ordering Information – ChiroSil Phases

ChiroSil Phase (5µm)	Column Dimensions (mm)			Guard Cartridge[1] (for 4.6mm i.d. columns)
	150 x 2.1	150 x 4.6	250 x 4.6	
RCA(+)	799003	799001	799002	799200
SCA(-)	799103	799101	799102	799100

[1] Use with guard cartridge holder 731441