

# 06

## Chiral Separation Columns and Packing Materials

CHIRAL ART-----	62-66
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## Polysaccharide type

### CHIRAL ART

- Applicable to various chiral compounds
- Applicable from LC/MS microanalysis to large-scale preparative purification
- Excellent resolution/durability
- High durability column that is suitable for SFC

- Particl size: 3, 5, 10, 20  $\mu\text{m}$
- USP L40, L51, L99, L119

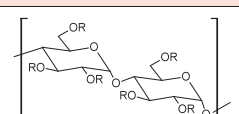
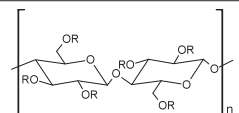
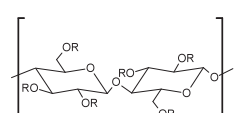
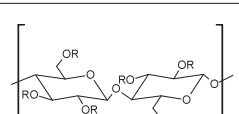
\*See pp.120-121 for details of SFC column.

#### HPLC columns/packing materials with polysaccharide derivatives as chiral selectors

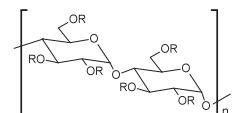
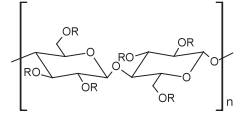
CHIRAL ART are HPLC columns/packing materials coated/immobilized with polysaccharide derivatives as chiral selector. CHIRAL ART immobilized type can be used either in normal- or reversed-phase modes. CHRAL ART are suitable for separations of wide range of chiral compounds, *cis-trans* isomers and geometric isomers. Packing materials are available in large quantities (multi kg).

### Specifications

#### Immobilized type

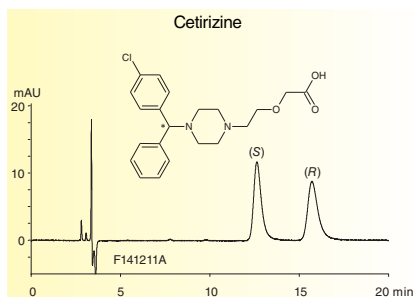
Column/Packing material	Particle size ( $\mu\text{m}$ )	Chiral selector	USP Classification
CHIRAL ART Amylose-SA	3 5 10 20	 Amylose tris(3,5-dimethylphenylcarbamate)	L99
CHIRAL ART Cellulose-SB		 Cellulose tris(3,5-dimethylphenylcarbamate)	–
CHIRAL ART Cellulose-SC		 Cellulose tris(3,5-dichlorophenylcarbamate)	L119
CHIRAL ART Cellulose-SJ		 Cellulose tris(4-methylbenzoate)	–
Usable mobile phase	Normal-phase	<i>n</i> -hexane, <i>n</i> -heptane, methanol, ethanol, 2-propanol, acetonitrile, ethyl acetate, tetrahydrofuran, chloroform, <i>t</i> -butyl methyl ether, etc.	
	Reversed-phase	acetonitrile, methanol, ethanol, 2-propanol, tetrahydrofuran, water, aqueous buffer, etc.	

#### Coated type

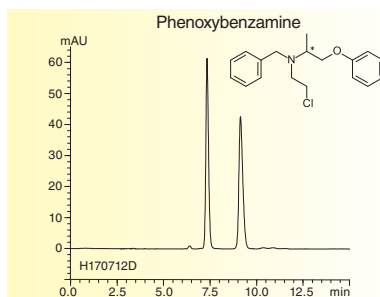
Column/Packing material	Particle size ( $\mu\text{m}$ )	Chiral selector	USP Classification
CHIRAL ART Amylose-C Neo	3 5 10 20	 Amylose tris(3,5-dimethylphenylcarbamate)	L51
CHIRAL ART Cellulose-C		 Cellulose tris(3,5-dimethylphenylcarbamate)	L40
Usable mobile phase	<i>n</i> -hexane, <i>n</i> -heptane, ethanol, 2-propanol, acetonitrile, etc.		

\*Inquire us for the Amylose-C

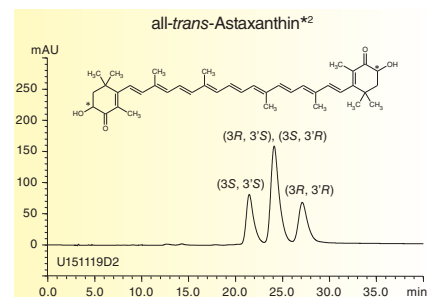
## Useful for separation of a wide range of chiral compounds



Column : CHIRAL ART Cellulose-C  
5  $\mu$ m, 250 X 4.6 mmI.D.  
Eluent : *n*-hexane/2-propanol/formic acid/DEA\*<sup>1</sup>  
(70/30/0.1/0.1)  
Flow rate : 1.0 mL/min  
Temperature : 25°C  
Detection : UV at 230 nm  
Injection : 2  $\mu$ L (0.2 mg/mL)



Column : CHIRAL ART Cellulose-SJ  
5  $\mu$ m, 250 X 4.6 mmI.D.  
Eluent : *n*-hexane/ethanol/diethylamine  
(95/5/0.1)  
Flow rate : 1.0 mL/min  
Temperature : 25°C  
Detection : UV at 270 nm  
Injection : 5  $\mu$ L (1 mg/mL)



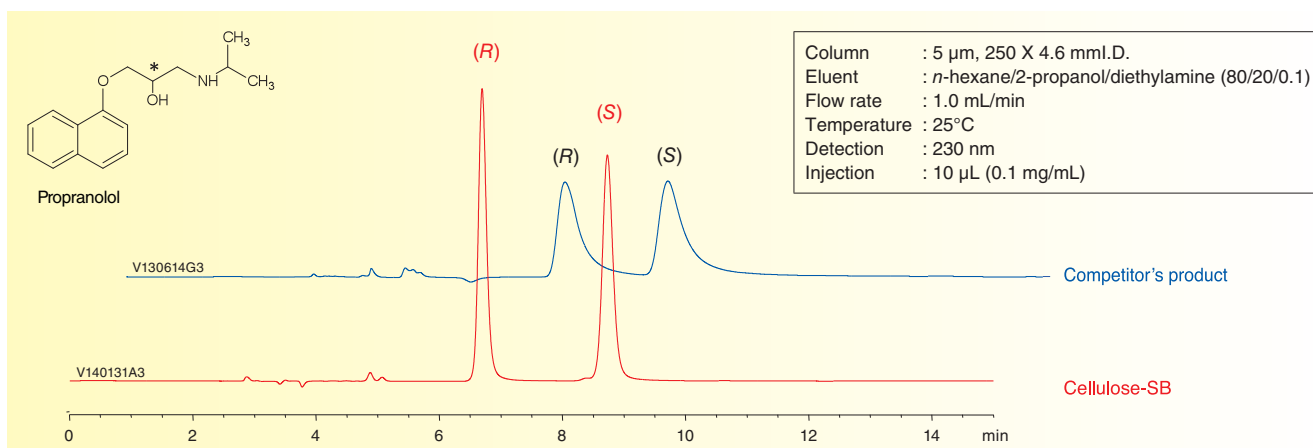
Column : CHIRAL ART Cellulose-SC  
5  $\mu$ m, 250 X 4.6 mmI.D.  
Eluent : *n*-hexane/THF (80/20)  
Flow rate : 1.0 mL/min  
Temperature : 25°C  
Detection : VIS at 476 nm  
Injection : 5  $\mu$ L (0.5 mg/mL)

\*<sup>2</sup>Courtesy of Fuji Chemical Industry Co., Ltd.

\*diethylamine

CHIRAL ART are suitable for separation of a wide range of compounds.

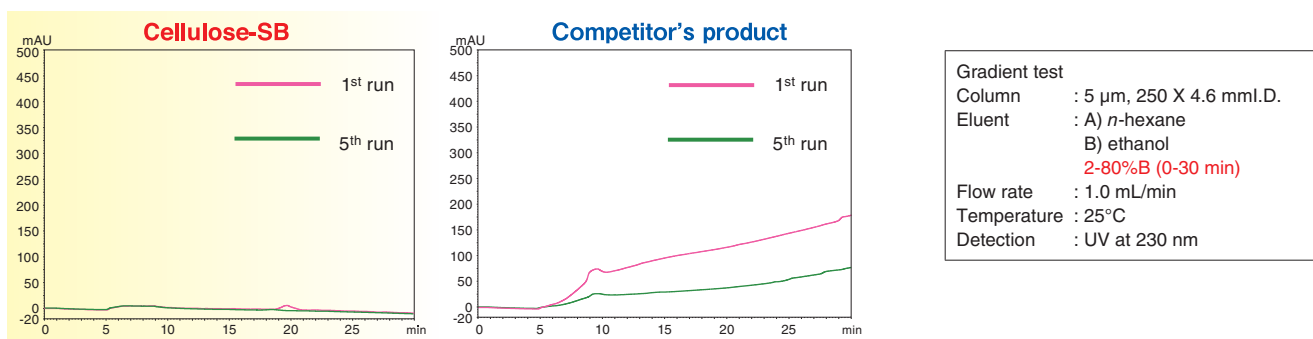
## Excellent peak shape



Column : 5  $\mu$ m, 250 X 4.6 mmI.D.  
Eluent : *n*-hexane/2-propanol/diethylamine (80/20/0.1)  
Flow rate : 1.0 mL/min  
Temperature : 25°C  
Detection : 230 nm  
Injection : 10  $\mu$ L (0.1 mg/mL)

CHIRAL ART provide good peak shapes on ionic and metal coordination compounds.

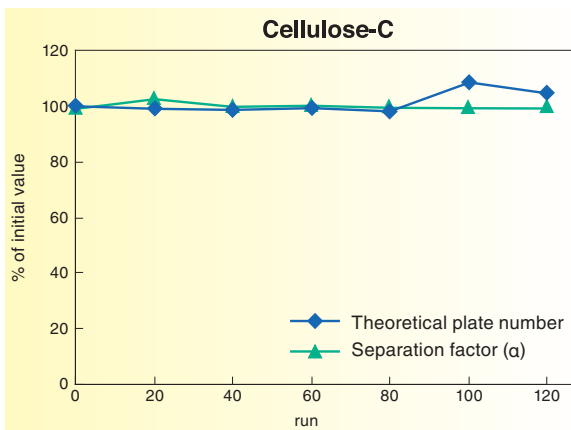
## Low column bleeding



Gradient test  
Column : 5  $\mu$ m, 250 X 4.6 mmI.D.  
Eluent : A) *n*-hexane  
B) ethanol  
2-80%B (0-30 min)  
Flow rate : 1.0 mL/min  
Temperature : 25°C  
Detection : UV at 230 nm

CHIRAL ART immobilized type show remarkably reduced background signal under the typical gradient conditions. CHIRAL ART immobilized type offer excellent robustness on gradient analysis and highly sensitive analysis on LC/MS due to the very low ion suppression as well as a stable baseline.

## Extended packing durability



### Sequential gradient test

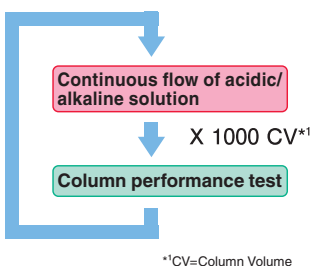
Column : 5  $\mu$ m, 250 X 4.6 mm.I.D.  
 Eluent : A) *n*-hexane, B) ethanol  
 0-100%B (0-15 min)  
 Flow rate : 3.0 mL/min  
 Pressure : 10-30 MPa/run  
 Temperature : 37°C

### Column performance test

Column : 5  $\mu$ m, 250 X 4.6 mm.I.D.  
 Eluent : *n*-hexane/ethanol (90/10)  
 Flow rate : 1.0 mL/min  
 Temperature : 37°C  
 Detection : UV at 230 nm  
 Sample : *trans*-Stilbene oxide

CHIRAL ART have outstanding packed bed stability provided by using high-strength super wide pore silica and innovative packing technology. The column efficiency and selectivity are maintained even after the sequential gradient tests at a high flow rate (three times higher than normal flow rate) and under high pressure (rapid pressure change). CHIRAL ART are useful for shortening analysis time, (re-)equilibration time, or/and column cleaning time by increasing the flow rate. CHIRAL ART are also effective when using highly viscose solvents as a mobile phase on immobilized type columns.

## Wide usable pH range (Immobilized type)



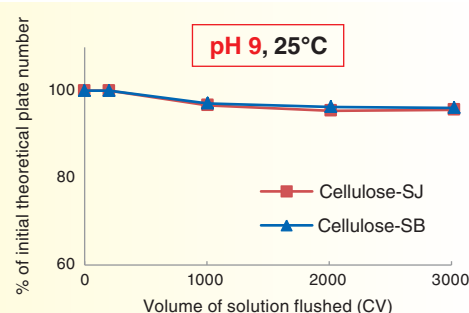
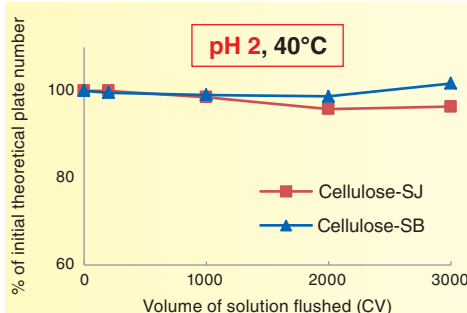
### Continuous flow of acidic/alkaline solution

Column : 5  $\mu$ m, 50 X 4.6 mm.I.D.  
 Eluent : Buffer/methanol (90/10)  
 Flow rate : 1.0 mL/min  
**[Acidic condition]**  
 Buffer : 0.1% H<sub>3</sub>PO<sub>4</sub> (pH 2)  
 Temperature : 40°C  
**[Alkaline condition]**  
 Buffer : 20 mM NH<sub>4</sub>HCO<sub>3</sub>-DEA\*<sup>2</sup> (pH 9)  
 Temperature : 25°C

\*CV=Column Volume  
<sup>2</sup>diethylamine

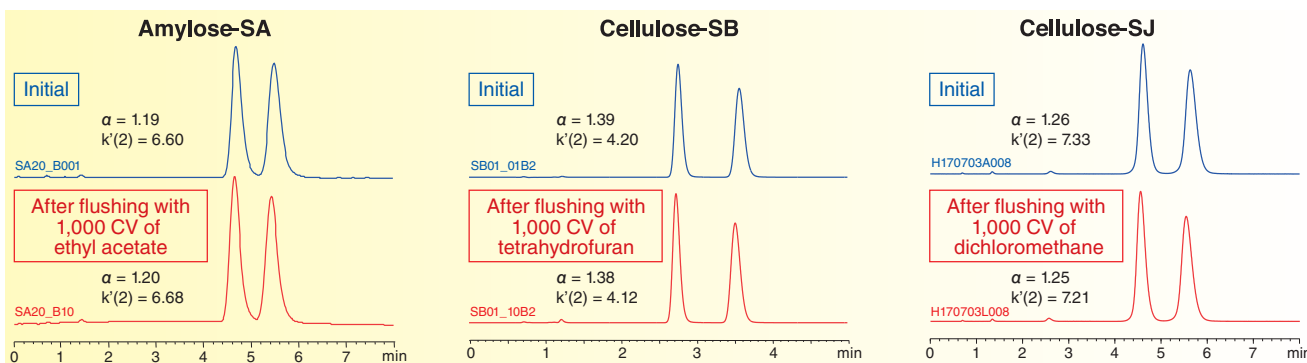
### Column performance test

Column : 5  $\mu$ m, 50 X 4.6 mm.I.D.  
 Eluent : methanol/water (70/30) for Cellulose-SJ  
 acetonitrile/water (30/70) for Cellulose-SB  
 Flow rate : 1.0 mL/min  
 Temperature : 25°C  
 Detection : UV at 254 nm  
 Sample : 1-(1-Naphthyl)ethanol for Cellulose-SJ  
 Benzoil for Cellulose-SB



CHIRAL ART immobilized type have excellent chemical stability and can be used across a wide pH range. CHIRAL ART also enable the robust analysis of ionic compounds requiring pH control of mobile phase in reversed-phase conditions.

## High solvent versatility (Immobilized type)



Retention rate of initial column performance (After flushing with 1,000 CV of each solvent at 40°C)

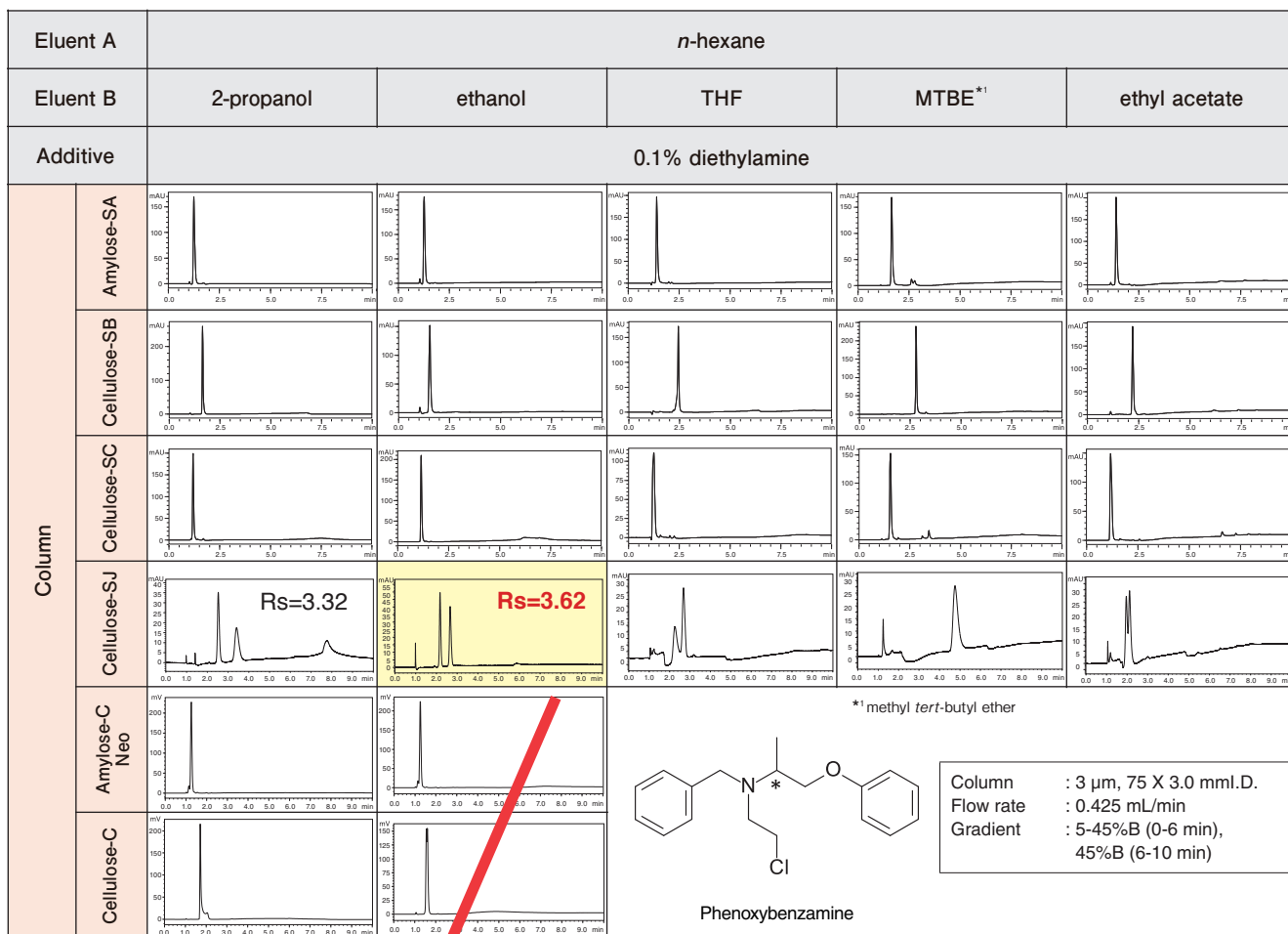
\*CV=Column Volume

	Amylose-SA		Cellulose-SB		Cellulose-SJ	
	$\alpha$	$k'(2)$	$\alpha$	$k'(2)$	$\alpha$	$k'(2)$
Ethyl acetate	100.3%	101.2%	100.0%	99.1%	99.3%	99.0%
Tetrahydrofuran	100.0%	100.0%	99.3%	98.0%	99.2%	99.7%
Dichloromethane	100.3%	100.6%	101.3%	99.6%	99.6%	98.4%

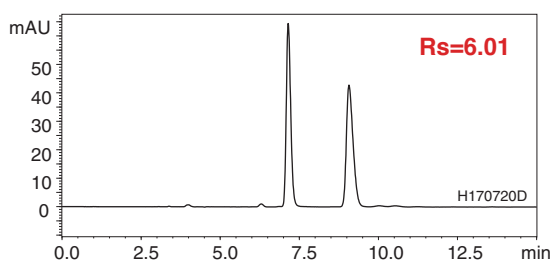
CHIRAL ART immobilized type have high solvent versatility. After flushing with various solvents, the losses of initial column performances were less than 2%.

## Method scouting

Method scouting is effective for method development of chiral compound separation. Rapid method development can be achieved by studying combination of mobile phases and columns comprehensively, and then optimizing a candidate condition.



optimization

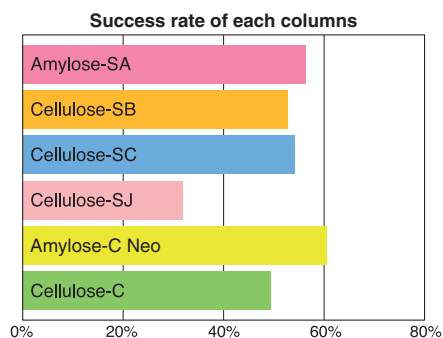


Column : CHIRAL ART Cellulose-SJ  
5 μm, 250 X 4.6 mm.I.D.  
Eluent : *n*-hexane/ethanol/diethylamine (95/5/0.1)  
Flow rate : 1.0 mL/min  
Temperature : 25°C  
Detection : UV at 270 nm  
Injection : 5 μL (1.0 mg/mL)

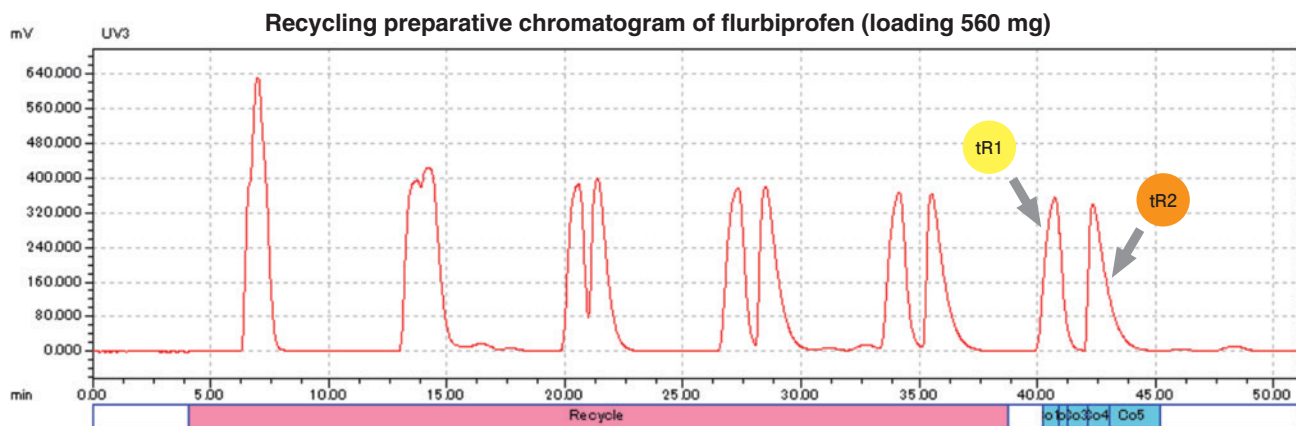
Based on the result of method scouting, the elution mode was changed to isocratic and the particle size and the column size were optimized.

### The result of screening using columns for chiral separation

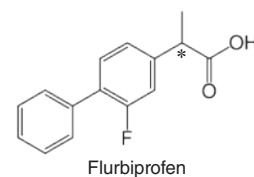
Success rate ; about 90% (Number of studies ; 520)



## High purity preparative purification of chiral compound using recycling HPLC



Column : CHIRAL ART Cellulose-C  
 5  $\mu$ m, 250 X 30 mm.I.D.  
 Eluent : *n*-hexane/2-propanol/TFA (95/5/0.1)  
 Flow rate : 45 mL/min  
 Detection : UV at 280 nm  
 Injection : 560 mg



	Single column		Recycling	
	tR1	tR2	tR1	tR2
Enantiomeric purity (%ee)	>99	>97	>99	99
Yield (%)	87	74	89	90
Productivity (mg product/hr)	122	103	335	336
Solvent consumption (L/g-product)	22	26	2.0	2.0

**Productivity : about 3 times higher**

**Solvent consumption : about 1/10**

Productivity of single column preparation is estimated based on the stacking injection of the sample every 2.5 minutes

Above is the result of preparative separation of chiral compound, flurbiprofen. Recycling preparation can achieve high purity and high yield on purification of the compound which is difficult to optimize preparative separation conditions using a single column method. Recycling preparation greatly contributes to reduction of solvent consumption and costs on purification as mobile phase is circulated during recycling mode.

## Synthesized macromolecule-type

# YMC CHIRAL NEA (R), (S)

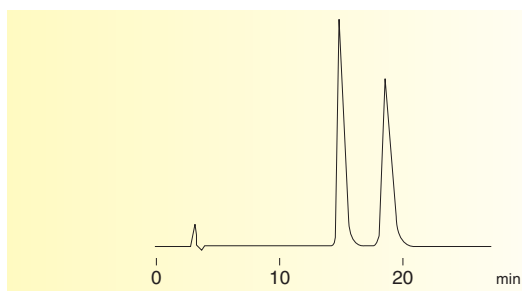
- Synthesized macromolecule-type chiral column
- Elution order can be reversed by selection (R) or (S)
- Can be used in both normal-phase and reversed-phase
- Available for bulk scale

- Particle size : 5  $\mu\text{m}$
- Pore size : 300  $\text{\AA}$
- Usable pH range : 2-6.5

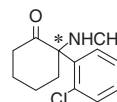
### Chiral polymer-bonded silica gel for chiral separation

YMC CHIRAL NEA (R) and (S) are chiral polymer-bonded silica gel for chiral separation. Chiral discrimination is based on the higher-order structure of chiral macromolecules, which includes hydrogen bonding,  $\pi$ - $\pi$  interaction, hydrophobic interaction, etc. YMC CHIRAL NEA (R) and (S) have excellent durability and cost performance.

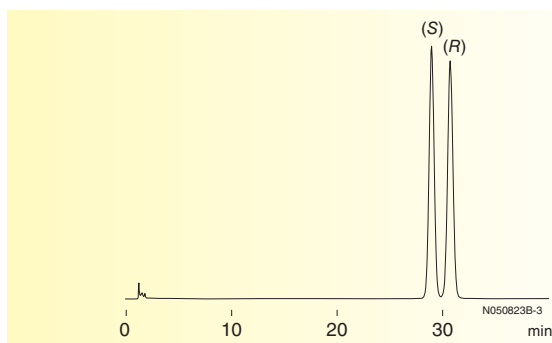
### For separation of chiral compounds



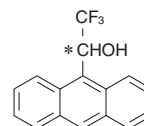
Ketamine



Column : YMC CHIRAL NEA (R)  
250 X 4.6 mm I.D.  
Eluent : acetonitrile/0.5 M NaClO<sub>4</sub> (40/60)  
Flow rate : 1.0 mL/min  
Temperature : ambient  
Detection : UV at 268 nm



2,2,2-Trifluoro-1-(9-anthryl) ethanol



Column : YMC CHIRAL NEA (R)  
250 X 4.6 mm I.D.  
Eluent : acetonitrile/water (40/60)  
Flow rate : 1.0 mL/min  
Temperature : 30°C  
Detection : UV at 254 nm

## Cyclodextrin-type

# YMC CHIRAL CD BR

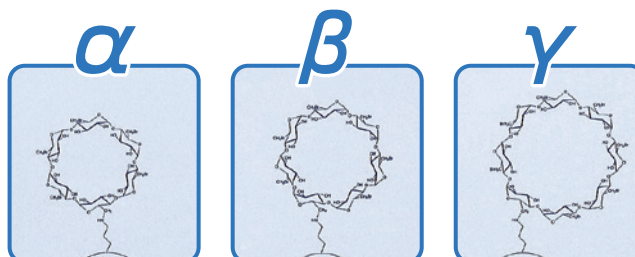
- Cyclodextrin type chiral separation column
- Useful for separation of optical isomers and structural isomers
- Three cavity types,  $\alpha$ ,  $\beta$  and  $\gamma$ , are available

- Particle size : 5  $\mu\text{m}$
- Pore size : 120  $\text{\AA}$
- Usable pH range : 3.5-6.5

### Chiral separation column utilizing host-guest interaction

YMC CHIRAL CD BR are composed of 3 types of chiral separation columns. Each column possesses  $\alpha$ -,  $\beta$ - or  $\gamma$ -bromo-cyclodextrin as a bonded phase. Selection from the 3 types of columns enables analysis of a wide range of compounds. In addition, YMC CHIRAL CD BR show different selectivity from ODS because the separation is based on host-guest interaction. YMC CHIRAL CD BR are useful for separating structural isomers that are difficult to separate on ODS.

Three types;  $\alpha$ ,  $\beta$  and  $\gamma$ -CD BR are available



Ordering Information -Columns-

CHIRAL ART

Particle size (µm)	Column size inner diameter X length (mm)	Immobilized type				Coated type	
		Amylose-SA	Cellulose-SB	Cellulose-SC	Cellulose-SJ	Amylose-C Neo	Cellulose-C
3	2.0 X 75	KSA99S03-L502WT	KSB99S03-L502WT	KSC99S03-L502WT	KSJ99S03-L502WT	KBN99S03-L502WT	KCN99S03-L502WT
	2.0 X 100	KSA99S03-1002WT	KSB99S03-1002WT	KSC99S03-1002WT	KSJ99S03-1002WT	KBN99S03-1002WT	KCN99S03-1002WT
	2.0 X 150	KSA99S03-1502WT	KSB99S03-1502WT	KSC99S03-1502WT	KSJ99S03-1502WT	KBN99S03-1502WT	KCN99S03-1502WT
	2.0 X 250	KSA99S03-2502WT	KSB99S03-2502WT	KSC99S03-2502WT	KSJ99S03-2502WT	KBN99S03-2502WT	KCN99S03-2502WT
	3.0 X 50	KSA99S03-0503WT	KSB99S03-0503WT	KSC99S03-0503WT	KSJ99S03-0503WT	KBN99S03-0503WT	KCN99S03-0503WT
	3.0 X 75	KSA99S03-L503WT	KSB99S03-L503WT	KSC99S03-L503WT	KSJ99S03-L503WT	KBN99S03-L503WT	KCN99S03-L503WT
	3.0 X 100	KSA99S03-1003WT	KSB99S03-1003WT	KSC99S03-1003WT	KSJ99S03-1003WT	KBN99S03-1003WT	KCN99S03-1003WT
	3.0 X 150	KSA99S03-1503WT	KSB99S03-1503WT	KSC99S03-1503WT	KSJ99S03-1503WT	KBN99S03-1503WT	KCN99S03-1503WT
	3.0 X 250	KSA99S03-2503WT	KSB99S03-2503WT	KSC99S03-2503WT	KSJ99S03-2503WT	KBN99S03-2503WT	KCN99S03-2503WT
	4.6 X 50	KSA99S03-0546WT	KSB99S03-0546WT	KSC99S03-0546WT	KSJ99S03-0546WT	KBN99S03-0546WT	KCN99S03-0546WT
	4.6 X 75	KSA99S03-L546WT	KSB99S03-L546WT	KSC99S03-L546WT	KSJ99S03-L546WT	KBN99S03-L546WT	KCN99S03-L546WT
	4.6 X 100	KSA99S03-1046WT	KSB99S03-1046WT	KSC99S03-1046WT	KSJ99S03-1046WT	KBN99S03-1046WT	KCN99S03-1046WT
4.6 X 150	KSA99S03-1546WT	KSB99S03-1546WT	KSC99S03-1546WT	KSJ99S03-1546WT	KBN99S03-1546WT	KCN99S03-1546WT	
4.6 X 250	KSA99S03-2546WT	KSB99S03-2546WT	KSC99S03-2546WT	KSJ99S03-2546WT	KBN99S03-2546WT	KCN99S03-2546WT	
5	4.6 X 150	KSA99S05-1546WT	KSB99S05-1546WT	KSC99S05-1546WT	KSJ99S05-1546WT	KBN99S05-1546WT	KCN99S05-1546WT
	4.6 X 250	KSA99S05-2546WT	KSB99S05-2546WT	KSC99S05-2546WT	KSJ99S05-2546WT	KBN99S05-2546WT	KCN99S05-2546WT
	10 X 250	KSA99S05-2510WT	KSB99S05-2510WT	KSC99S05-2510WT	KSJ99S05-2510WT	KBN99S05-2510WT	KCN99S05-2510WT
	20 X 250	KSA99S05-2520WX	KSB99S05-2520WX	KSC99S05-2520WX	KSJ99S05-2520WX	KBN99S05-2520WX	KCN99S05-2520WX
	30 X 250	KSA99S05-2530WX	KSB99S05-2530WX	KSC99S05-2530WX	KSJ99S05-2530WX	KBN99S05-2530WX	KCN99S05-2530WX

CHIRAL ART Guard cartridges

Particle size (µm)	Column size inner diameter X length (mm)	Immobilized type				Coated type	
		Amylose-SA	Cellulose-SB	Cellulose-SC	Cellulose-SJ	Amylose-C Neo	Cellulose-C
3	2.1 X 10	KSA99S03-01Q1GC	KSB99S03-01Q1GC	KSC99S03-01Q1GC	KSJ99S03-01Q1GC	KBN99S03-01Q1GC	KCN99S03-01Q1GC
	3.0 X 10	KSA99S03-0103GC	KSB99S03-0103GC	KSC99S03-0103GC	KSJ99S03-0103GC	KBN99S03-0103GC	KCN99S03-0103GC
	4.0 X 10	KSA99S03-0104GC	KSB99S03-0104GC	KSC99S03-0104GC	KSJ99S03-0104GC	KBN99S03-0104GC	KCN99S03-0104GC
5	4.0 X 10	KSA99S05-0104GC	KSB99S05-0104GC	KSC99S05-0104GC	KSJ99S05-0104GC	KBN99S05-0104GC	KCN99S05-0104GC
	10 X 10	KSA99S05-0110CC	KSB99S05-0110CC	KSC99S05-0110CC	KSJ99S05-0110CC	KBN99S05-0110CC	KCN99S05-0110CC

\*Guard cartridge holder required, part no. XPGCH-Q1 for 2.1-4.0 mm.I.D. and XPCHSPW1 for 10 mm.I.D.

Alcyon SFC CSP

Particle size (µm)	Column size inner diameter X length (mm)	Immobilized type				Coated type	
		Amylose-SA	Cellulose-SB	Cellulose-SC	Cellulose-SJ	Amylose-C Neo	Cellulose-C
3	2.1 X 150	KSA99S03-15Q1WTS	KSB99S03-15Q1WTS	KSC99S03-15Q1WTS	KSJ99S03-15Q1WTS	KBN99S03-15Q1WTS	KCN99S03-15Q1WTS
	3.0 X 50	KSA99S03-0503WTS	KSB99S03-0503WTS	KSC99S03-0503WTS	KSJ99S03-0503WTS	KBN99S03-0503WTS	KCN99S03-0503WTS
	3.0 X 100	KSA99S03-1003WTS	KSB99S03-1003WTS	KSC99S03-1003WTS	KSJ99S03-1003WTS	KBN99S03-1003WTS	KCN99S03-1003WTS
	3.0 X 150	KSA99S03-1503WTS	KSB99S03-1503WTS	KSC99S03-1503WTS	KSJ99S03-1503WTS	KBN99S03-1503WTS	KCN99S03-1503WTS
	4.6 X 150	KSA99S03-1546WTS	KSB99S03-1546WTS	KSC99S03-1546WTS	KSJ99S03-1546WTS	KBN99S03-1546WTS	KCN99S03-1546WTS
4.6 X 250	KSA99S03-2546WTS	KSB99S03-2546WTS	KSC99S03-2546WTS	KSJ99S03-2546WTS	KBN99S03-2546WTS	KCN99S03-2546WTS	
5	2.1 X 150	KSA99S05-15Q1WTS	KSB99S05-15Q1WTS	KSC99S05-15Q1WTS	KSJ99S05-15Q1WTS	KBN99S05-15Q1WTS	KCN99S05-15Q1WTS
	4.6 X 150	KSA99S05-1546WTS	KSB99S05-1546WTS	KSC99S05-1546WTS	KSJ99S05-1546WTS	KBN99S05-1546WTS	KCN99S05-1546WTS
	4.6 X 250	KSA99S05-2546WTS	KSB99S05-2546WTS	KSC99S05-2546WTS	KSJ99S05-2546WTS	KBN99S05-2546WTS	KCN99S05-2546WTS
	10 X 250	KSA99S05-2510WTS	KSB99S05-2510WTS	KSC99S05-2510WTS	KSJ99S05-2510WTS	KBN99S05-2510WTS	KCN99S05-2510WTS
	20 X 250	KSA99S05-2520WTS	KSB99S05-2520WTS	KSC99S05-2520WTS	KSJ99S05-2520WTS	KBN99S05-2520WTS	KCN99S05-2520WTS

\*See pp.120-121 for details of Alcyon SFC CSP

Ordering Information -Packing Materials-

CHIRAL ART

Particle size (µm)	Immobilized type				Coated type	
	Amylose-SA	Cellulose-SB	Cellulose-SC	Cellulose-SJ	Amylose-C Neo	Cellulose-C
5	KSA99S05	KSB99S05	KSC99S05	KSJ99S05	KBN99S05	KCN99S05
10	KSA99S11	KSB99S11	KSC99S11	KSJ99S11	KBN99S11	KCN99S11
20	KSA99S21	KSB99S21	KSC99S21	KSJ99S21	KBN99S21	KCN99S21

\*Inquire us for the Amylose-C



## Ordering Information -Columns-

### YMC CHIRAL NEA(R)(S) : Reversed-phase

Phase dimension	Column I.D. (mm)	Column length (mm)		Guard cartridges	
		150	250	I.D. (mm)	10 mm length
NEA(R) 300 Å 5 μm	4.6	NR30S05-1546WT	NR30S05-2546WT	4.0	NR30S05-0104GC
NEA(S) 300 Å 5 μm	4.6	NS30S05-1546WT	NS30S05-2546WT	4.0	NS30S05-0104GC

### YMC CHIRAL NEA(R)(S) : Normal-phase

Phase dimension	Column I.D. (mm)	Column length (mm)		Guard cartridges	
		150	250	I.D. (mm)	10 mm length
NEA(R) 300 Å 5 μm	4.6	CR30S05-1546WT	CR30S05-2546WT	4.0	CR30S05-0104GC
NEA(S) 300 Å 5 μm	4.6	CS30S05-1546WT	CS30S05-2546WT	4.0	CS30S05-0104GC

### YMC CHIRAL CD BR

Phase dimension	Column I.D. (mm)	Column length (mm)		Guard cartridges	
		150	250	I.D. (mm)	10 mm length
α-CD BR 120 Å 5 μm	4.6	DA12S05-1546WT	DA12S05-2546WT	4.0	DA12S05-0104GC
β-CD BR 120 Å 5 μm	4.6	DB12S05-1546WT	DB12S05-2546WT	4.0	DB12S05-0104GC
γ-CD BR 120 Å 5 μm	4.6	DG12S05-1546WT	DG12S05-2546WT	4.0	DG12S05-0104GC

\*Guard cartridge holder required, part no. XPGCH-Q1.