

06

Chiral Separation Columns and Packing Materials

| | |
|------------------------------|-------|
| CHIRAL ART----- | 62-66 |
| YMC CHIRAL NEA (R), (S)----- | 67 |
| YMC CHIRAL CD BR----- | 67 |
| Ordering Information----- | 68-69 |

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 μm
- USP L40, L51, L99, L119

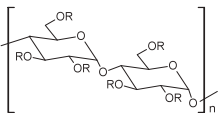
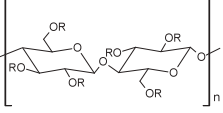
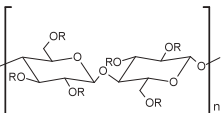
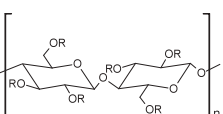
*See pp.120-121 for details of SFC column.

HPLC columns/packing materials with polysaccharide derivatives as chiral selectors

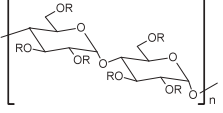
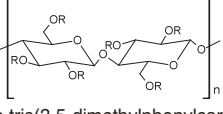
CHIRAL ART are HPLC columns/packing materials coated/immobilized with polysaccharide derivatives as chiral selector. CHIRAL ART immobilized type can be used either in normal- or reversed-phase modes. CHRAL ART are suitable for separations of wide range of chiral compounds, *cis-trans* isomers and geometric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

Specifications

Immobilized type

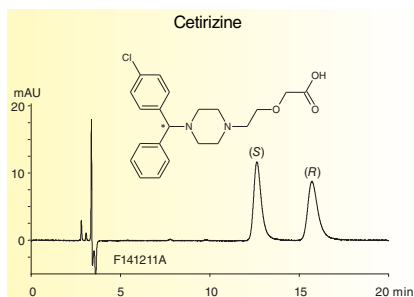
| Column/Packing material | Particle size (μm) | Chiral selector | USP Classification |
|-------------------------|--------------------|--|--------------------|
| CHIRAL ART Amylose-SA | 3 5 10 20 |  Amylose tris(3,5-dimethylphenylcarbamate) | L99 |
| CHIRAL ART Cellulose-SB | |  Cellulose tris(3,5-dimethylphenylcarbamate) | — |
| 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 (μm) | 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. | | |

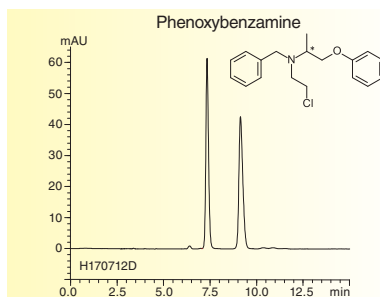
*Inquire us for the Amylose-C

Useful for separation of a wide range of chiral compounds

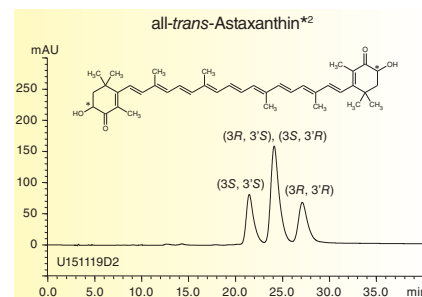


Column : CHIRAL ART Cellulose-C
5 μ m, 250 X 4.6 mmI.D.
Eluent : *n*-hexane/2-propanol/formic acid/DEA*¹
(70/30/0.1/0.1)
Flow rate : 1.0 mL/min
Temperature : 25°C
Detection : UV at 230 nm
Injection : 2 μ L (0.2 mg/mL)

*diethylamine



Column : CHIRAL ART Cellulose-SJ
5 μ 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 μ L (1 mg/mL)

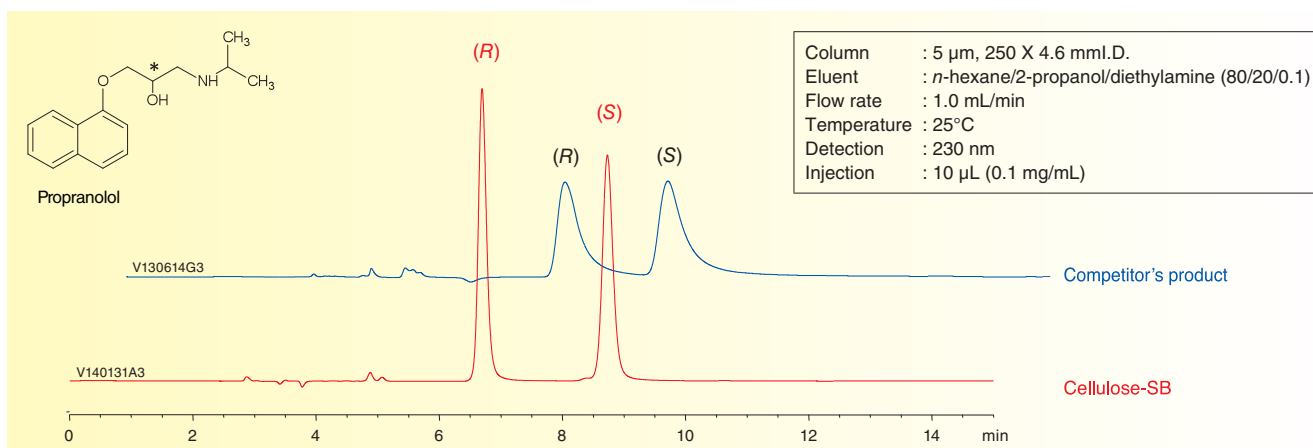


Column : CHIRAL ART Cellulose-SC
5 μ m, 250 X 4.6 mmI.D.
Eluent : *n*-hexane/THF (80/20)
Flow rate : 1.0 mL/min
Temperature : 25°C
Detection : VIS at 476 nm
Injection : 5 μ L (0.5 mg/mL)

*²Courtesy of Fuji Chemical Industry Co., Ltd.

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

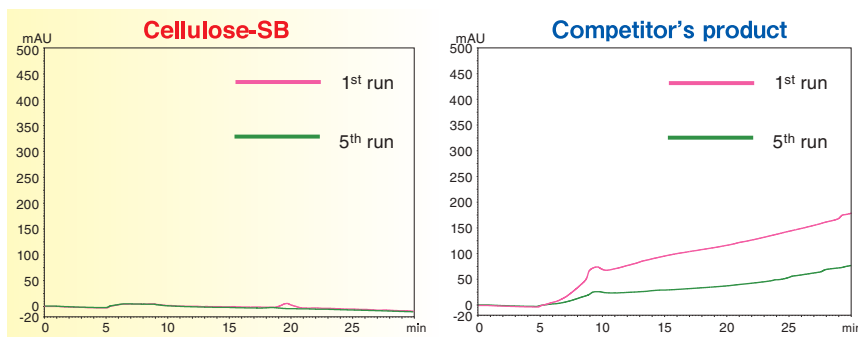
Excellent peak shape



Column : 5 μ 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 μ L (0.1 mg/mL)

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

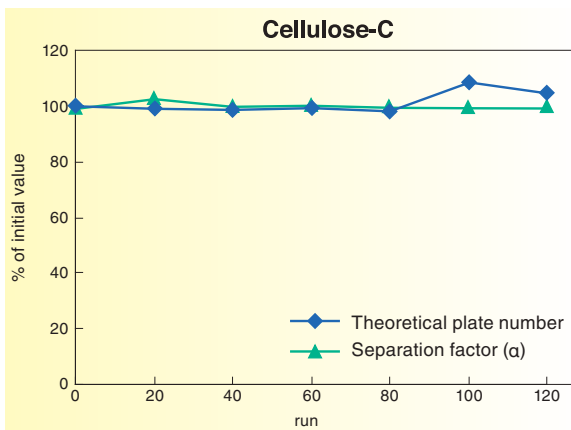
Low column bleeding



Gradient test
Column : 5 μ 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

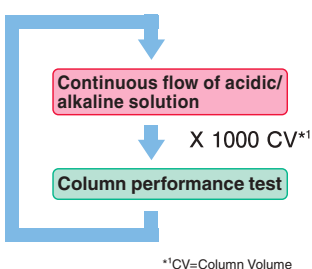
Column : 5 μ m, 250 X 4.6 mm.I.D.
 Eluent : A) *n*-hexane, B) ethanol
 0-100%B (0-15 min)
 Flow rate : 3.0 mL/min
 Pressure : 10-30 MPa/run
 Temperature : 37°C

Column performance test

Column : 5 μ 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)



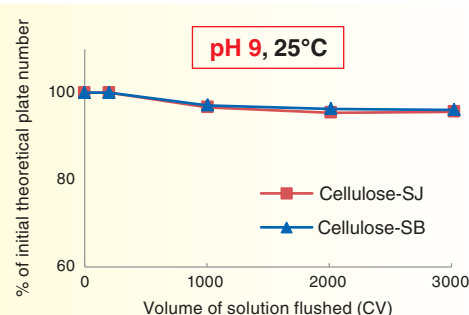
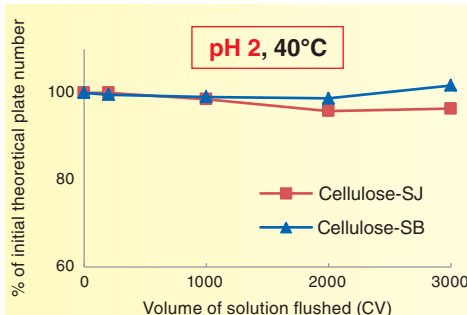
Continuous flow of acidic/alkaline solution

Column : 5 μ m, 50 X 4.6 mm.I.D.
 Eluent : Buffer/methanol (90/10)
 Flow rate : 1.0 mL/min
[Acidic condition]
 Buffer : 0.1% H₃PO₄ (pH 2)
 Temperature : 40°C
[Alkaline condition]
 Buffer : 20 mM NH₄HCO₃-DEA*² (pH 9)
 Temperature : 25°C

*CV=Column Volume
²diethylamine

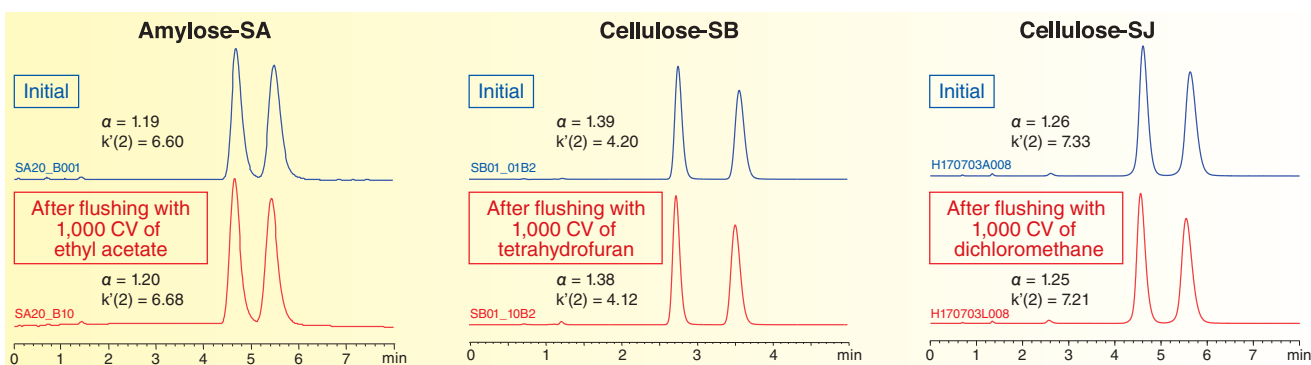
Column performance test

Column : 5 μ 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)

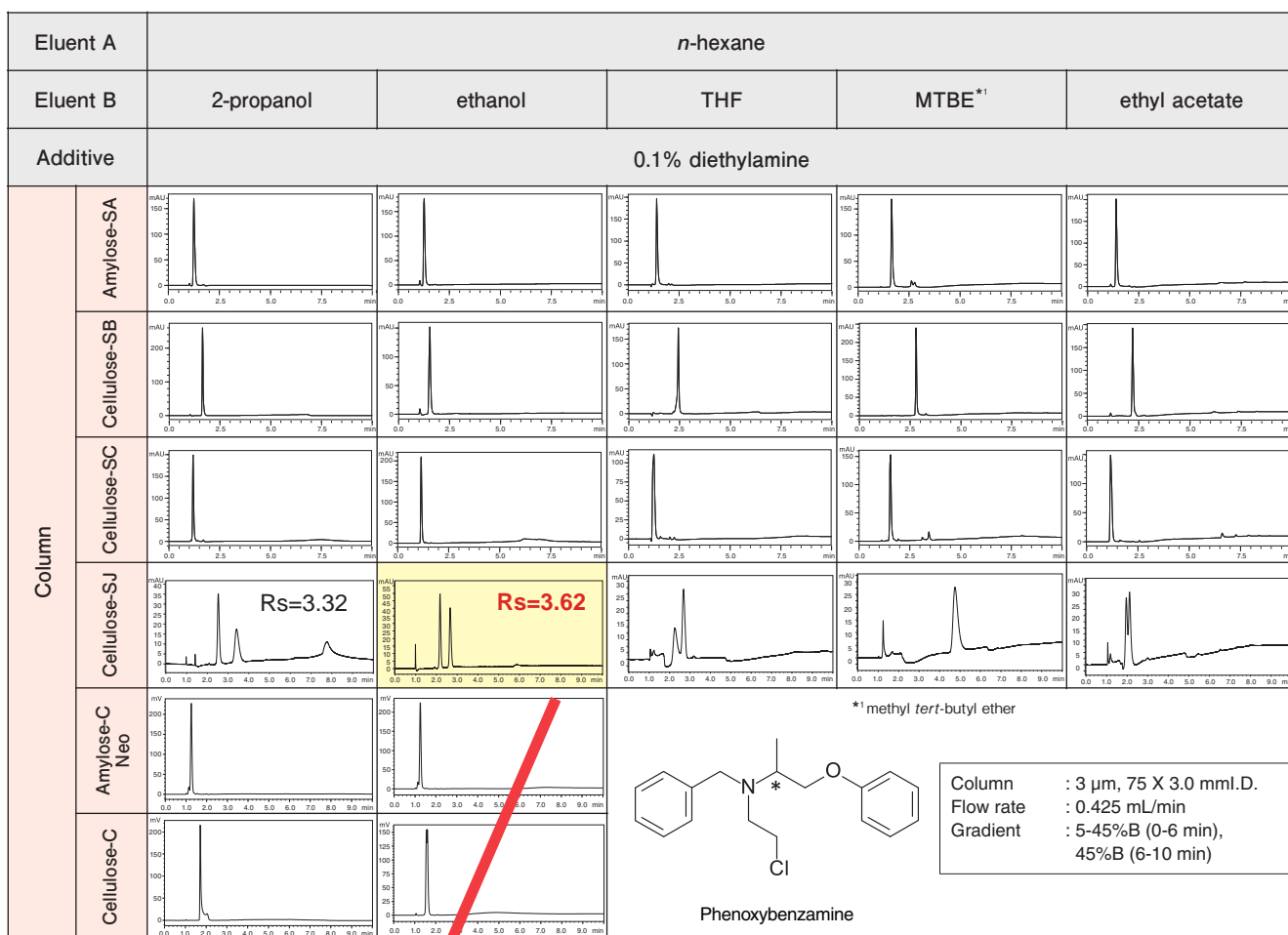
*CV=Column Volume

| | Amylose-SA | | Cellulose-SB | | Cellulose-SJ | |
|-----------------|------------|---------|--------------|---------|--------------|---------|
| | α | $k'(2)$ | α | $k'(2)$ | α | $k'(2)$ |
| 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% |

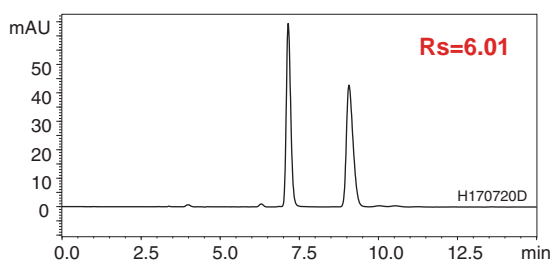
CHIRAL ART immobilized type have high solvent versatility. After flushing with various solvents, the losses of initial column performances were less than 2%.

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

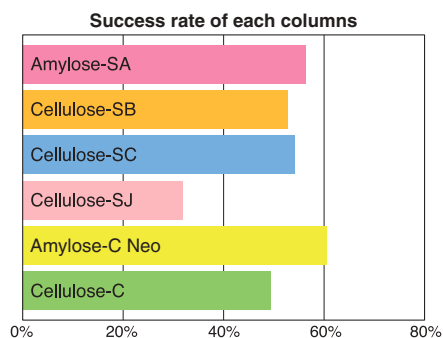


| | |
|-------------|--|
| Column | : CHIRAL ART Cellulose-SJ |
| | 5 μm, 250 X 4.6 mm.I.D. |
| Eluent | : <i>n</i> -hexane/ethanol/diethylamine (95/5/0.1) |
| Flow rate | : 1.0 mL/min |
| Temperature | : 25°C |
| Detection | : UV at 270 nm |
| Injection | : 5 μL (1.0 mg/mL) |

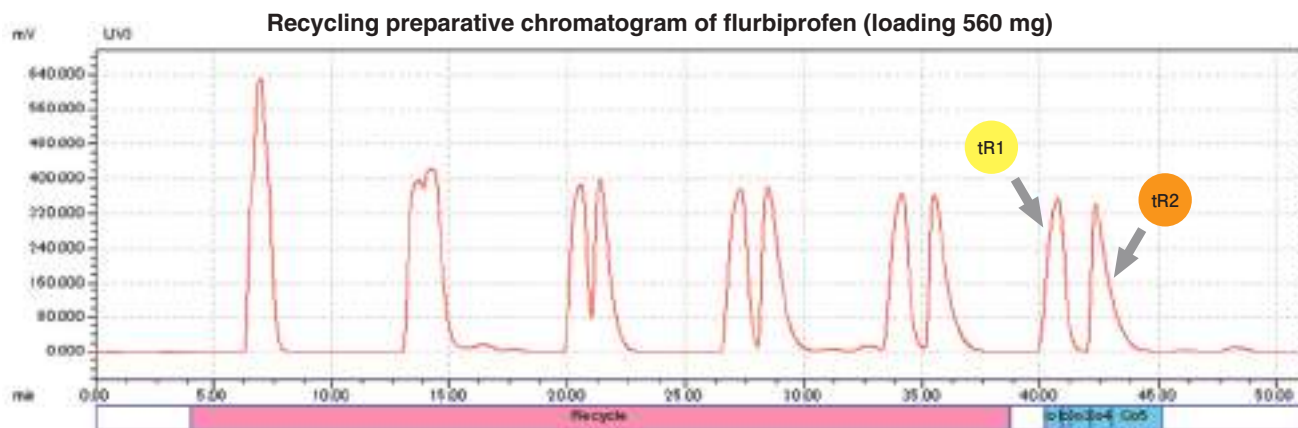
Based on the result of method scouting, the elution mode was changed to isocratic and the particle size and the column size were optimized.

The result of screening using columns for chiral separation

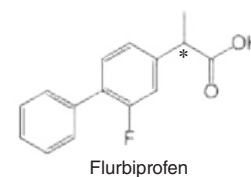
Success rate ; about 90% (Number of studies ; 520)



High purity preparative purification of chiral compound using recycling HPLC



Column : CHIRAL ART Cellulose-C
 5 μ 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)

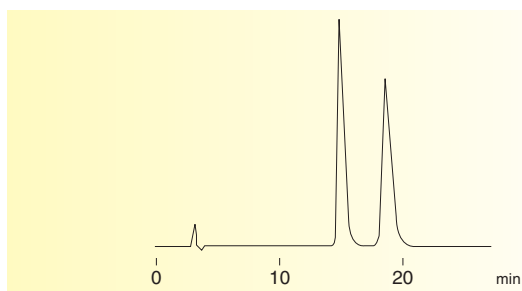
- Synthesized macromolecule-type chiral column
- Elution order can be reversed by selection (R) or (S)
- Can be used in both normal-phase and reversed-phase
- Available for bulk scale

- Particle size : 5 μm
- Pore size : 300 \AA
- Usable pH range : 2-6.5

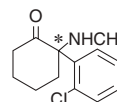
Chiral polymer-bonded silica gel for chiral separation

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, π - π interaction, hydrophobic interaction, etc. YMC CHIRAL NEA (R) and (S) have excellent durability and cost performance.

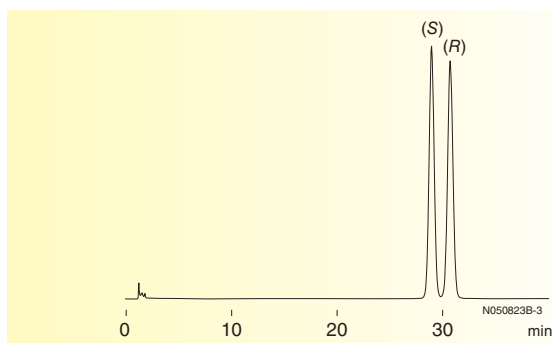
For separation of chiral compounds



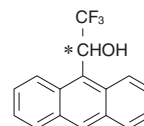
Ketamine



| | |
|-------------|---|
| Column | : YMC CHIRAL NEA (R) 250 X 4.6 mm I.D. |
| Eluent | : acetonitrile/0.5 M NaClO ₄ (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

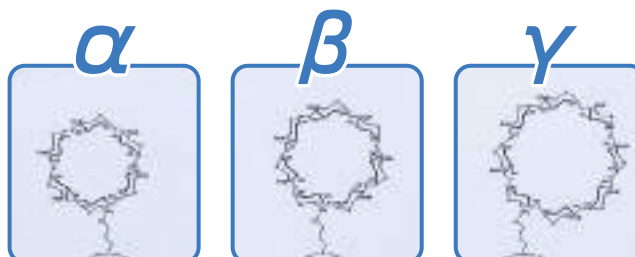
- Cyclodextrin type chiral separation column
- Useful for separation of optical isomers and structural isomers
- Three cavity types, α , β and γ , are available

- Particle size : 5 μm
- Pore size : 120 \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 α -, β - or γ -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; α , β and γ -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 |
| 5 | 4.6 X 250 | KSA99S03-2546WTS | KSB99S03-2546WTS | KSC99S03-2546WTS | KSJ99S03-2546WTS | KBN99S03-2546WTS | KCN99S03-2546WTS |
| | 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.