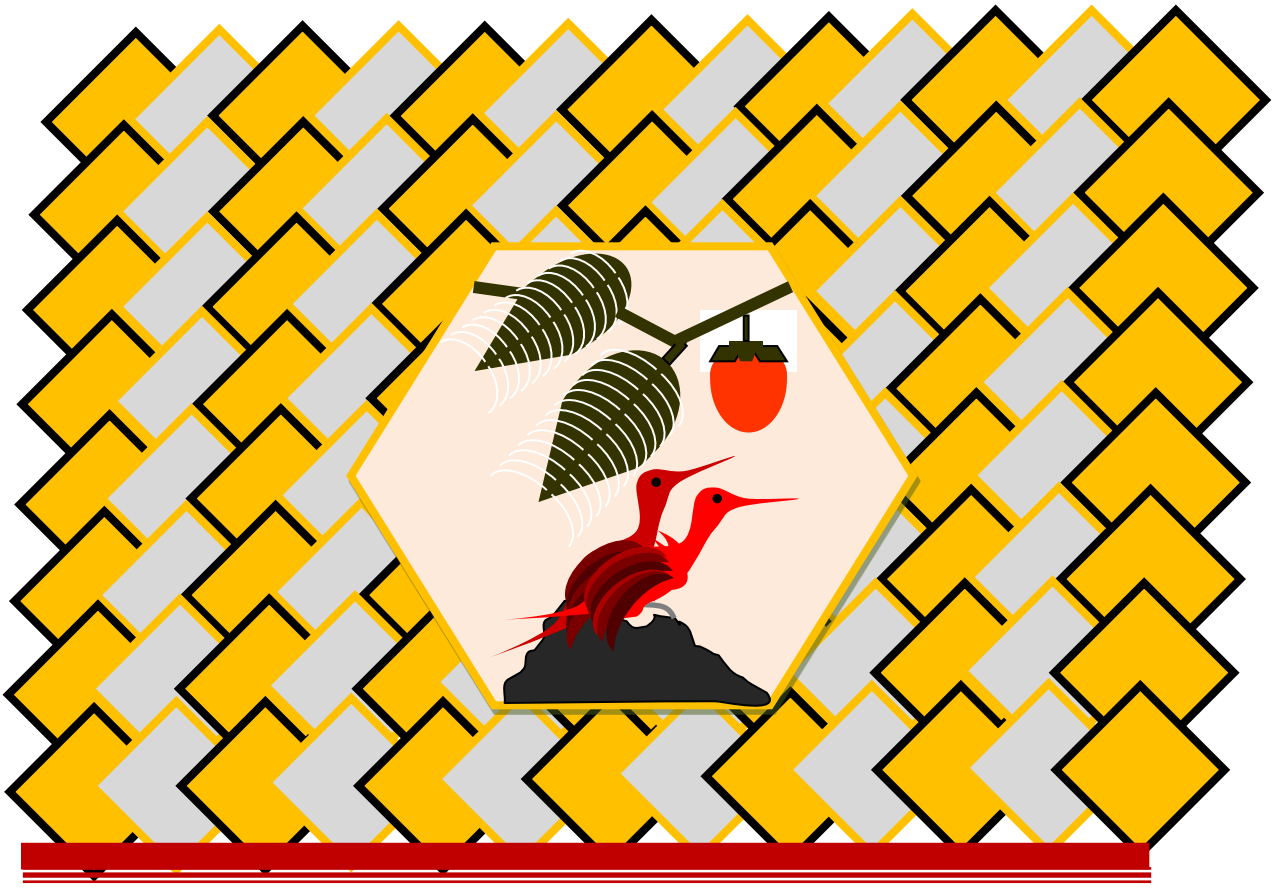
Technical Bulletin

CHROMATOREX

Amino Silica Gels

For Sugar Separation

NH-SG Silica



FUJI SILYSIA CHEMICAL LTD

Introduction

Sugar analysis and separations is broad application field in liquid chromatography. Up today, most separation media are developed as analytical grade only. Fuji Silysia chemical now developed new Amino-Type silica gel for preparative chromatography.
General NH type silica gel makes various separation problems for sugar separation when it is used under high amount of water condition. The pH of NH grade is too high (About 9.0). The higher pH makes denaturation of target compounds and damage of silica surface. Considering these problems, we developed a new grade, called SG (Sugar Grade) Amino silica. SG grade will minimize or avoid such a problem and enable to separate various sugar compounds easily.

Chromatorex Physical Properties

There are 3 major grades to cover preparative applications:

- NH MB100-75/200 Open column
- NH MB100-40/75 Flash column, Cartridge column
- NH SMB100-20/45 Cartridge column, HPLC

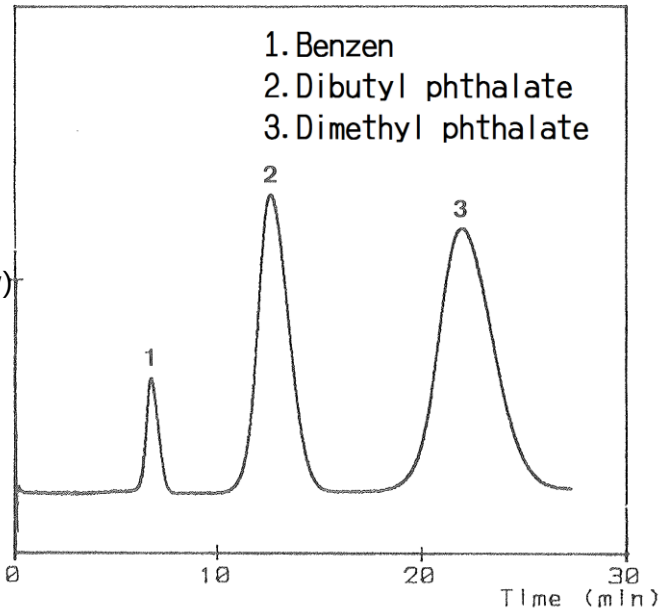
The table shows typical physical properties of NH SG silica gel. These grades are bonded with amino silane and neutral pH:

Items	**NH SMB100-20/45SG**	**NH MB100-40/75SG**	**NH MB100-75/200SG**
Base Silica gel			
Surface area (m^2/g)	300	300	300
Pore volume (ml/g)	0.80	0.75	0.75
particle size (μm)	30	60	110
NH SG Grade			
Bulk density (g/ml)	0.63	0.63	0.63
pH (5% slurry)	7.5	7.5	7.5

Separation of Neutral Compounds

NH SG Silica grade shows similar separation of neutral compounds as NH silica.

Chromatography Operating Conditions:
Column: 20 x 360 mm glass column
Silica: **NH MB100-75/200SG (50g)**
Mobile phase: Ethylacetate/n-hexane 10/90 (w/w)
Flow rate: 16 ml/min
Detection: UV-254 nm
Samples: 1. Benzene
2. Dibutyl phthalate
3. Dimethyl phthalate



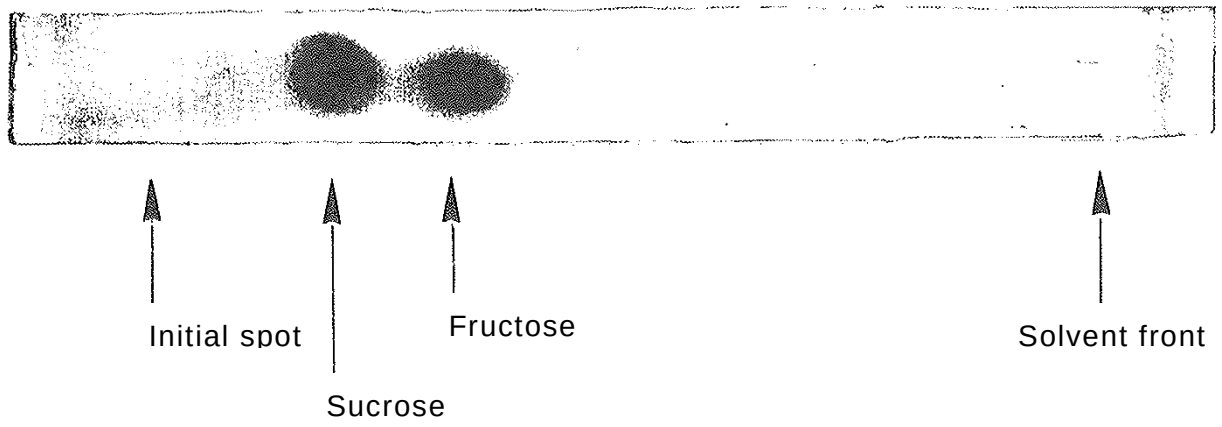
Separation of Sugar Compounds

It is well known that amino functional groups react with ketone and/or carboxyl group. NH SG silica gel can separate several sugar compounds under organic/H2O solvent conditions without showing such a chemical reaction. NH SG grade is used in normal phase conditions, especially, Hydrophilic Interaction Chromatography mode (HILIC). As typical HILIC mode, elution speed can be increased when % of water in the organic solvent is increased. Followings are several separation application data with acetonitrile / water solvent condition.

Pretest by TLC and HPLC

It is better to do pretest before using NH MB100-75/200SG.
NH TLC and NH2 SPS100-5SG is available for this test.

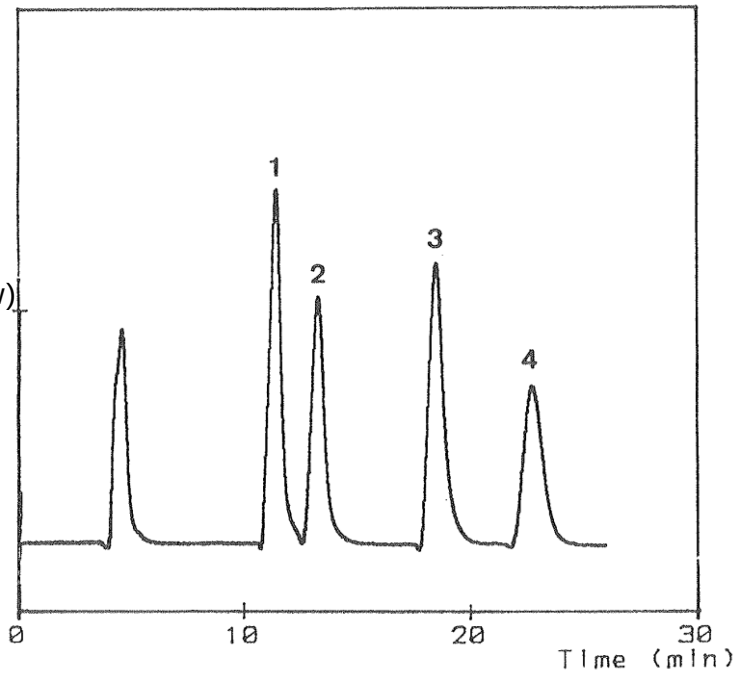
Separation by NH TLC (Acetonitrile/H2O 75/25(w/w))



Separation by HPLC

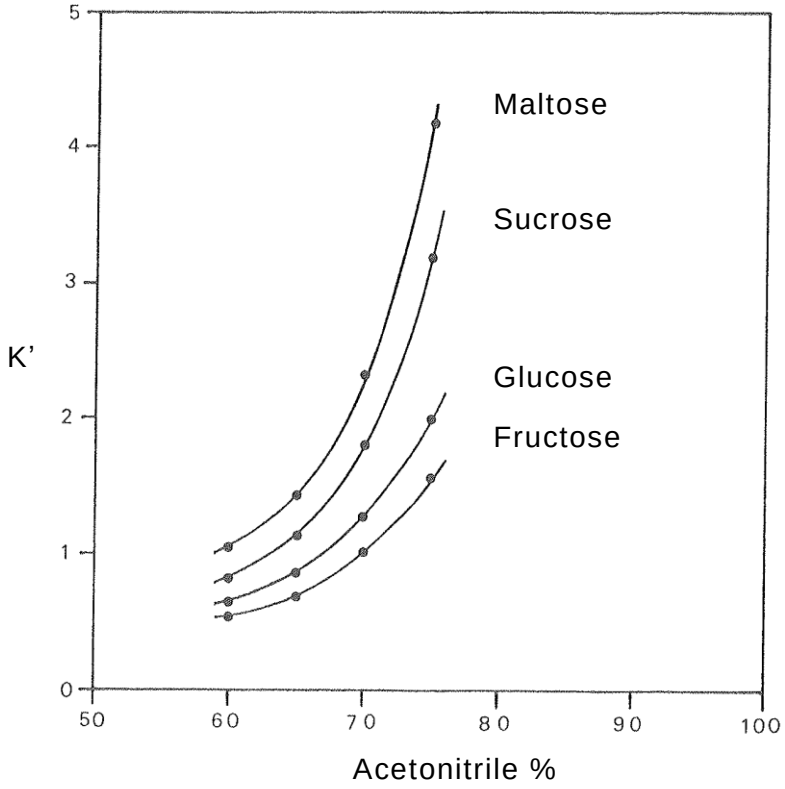
Chromatography Operating Conditions:

Column: 4.6x250mm HPLC column
Silica: NH SPS100-5SG
Mobile phase: Acetonitrile/H2O 75/25(w/w)
Flow rate: 1 ml/min
Detection: RI detector
Samples: 1. Fructose
2. Glucose
3. Sucrose
4. Maltose



Solvent conditions

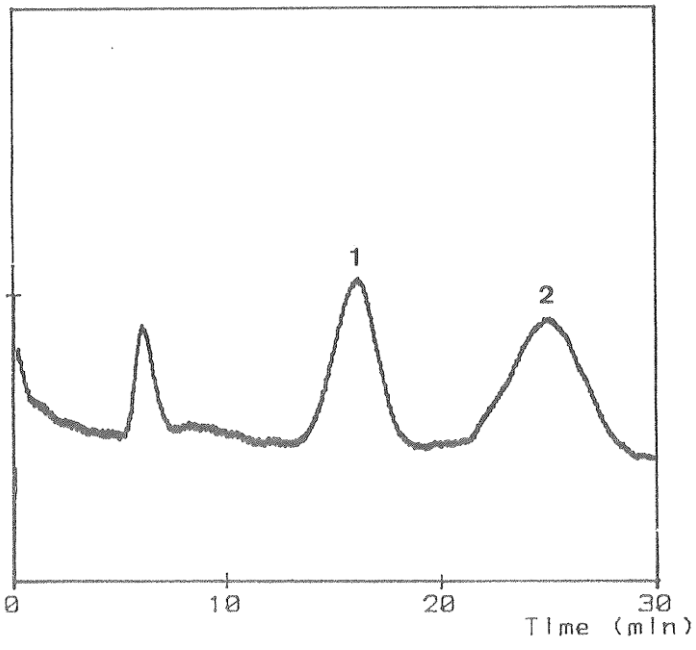
Solvents for sugar separation are normally Acetonitrile / H2O. Retention K' increases when the ratio of acetonitrile increases. The increase of acetonitrile should be controlled carefully, because solubility decreases when acetonitrile increases.



Application

(1) Sugar separation of monosaccharide and disaccharide

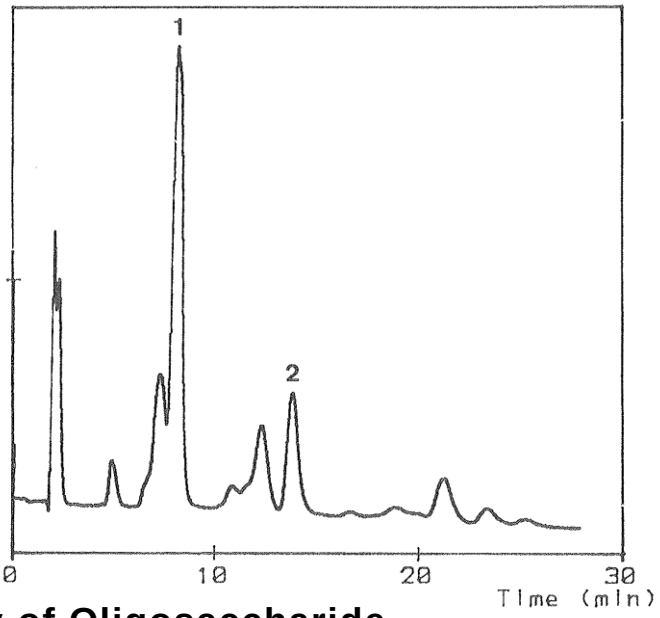
Column: 20 x 360 mm glass column
Silica: NH MB100-75/200SG (50g)
Mobile phase: Acetonitrile/H2O 75/25(w/w)
Pressure: 44 kPa
Flow rate: 16 ml/min
Detection: RI detector
Samples 1. Fructose
2. Sucrose



(2) Sugar separation of Oligosaccharide

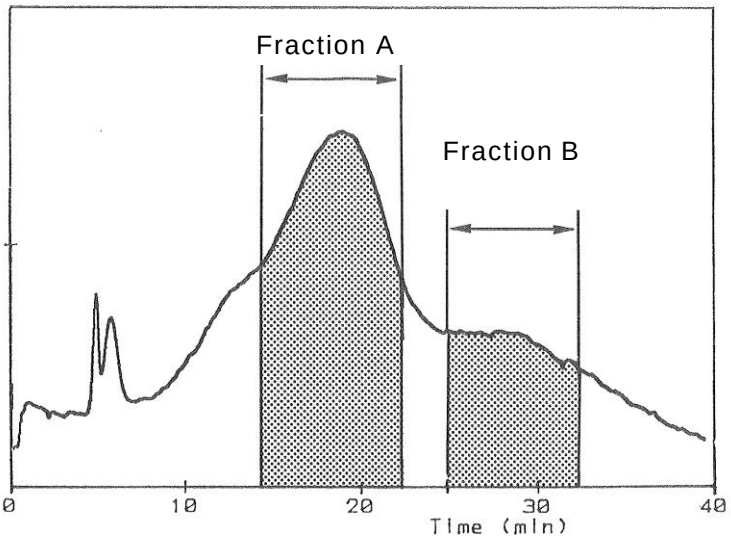
Pretest by HPLC

Column: 4.6x250mm HPLC column
Silica: NH SPS100-5SG
Mobile phase: Acetonitrile/H2O 70/30(w/w)
Flow rate: 2 ml/min
Detection: RI detector
Samples: 1. Isomaltose
2. Isomaltotriose

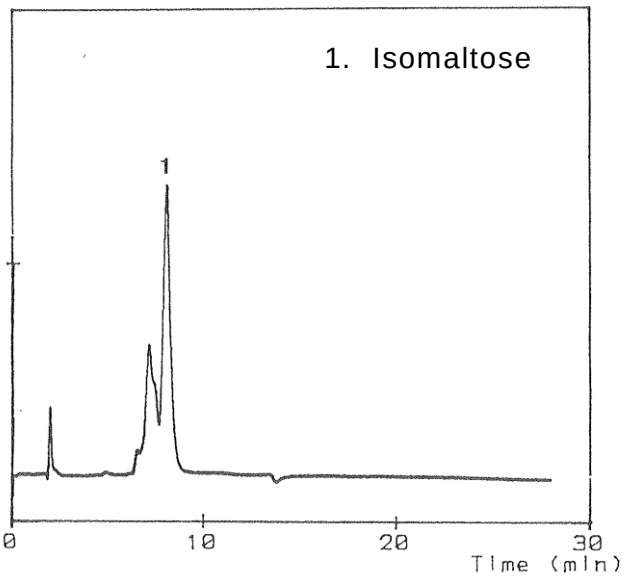


(2-1) Preparative chromatography of Oligosaccharide

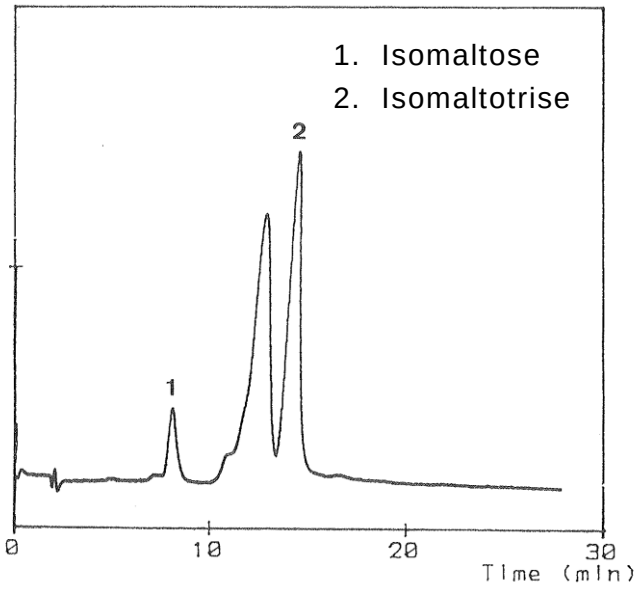
Column: 20 x 360 mm glass column
Silica: NH MB100-75/200SG (50g)
Mobile phase: Acetonitrile/H2O 70/30(w/w)
Pressure: 73 kPa
Flow rate: 16 ml/min
Detection: RI detector



(2-2) Fraction A



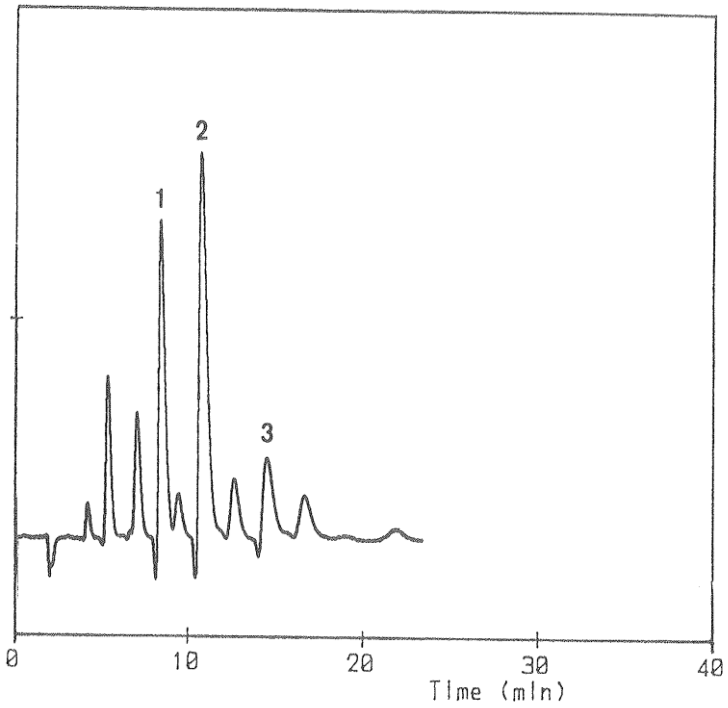
(2-3) Fraction B



(3) Separation of cyclodextrin

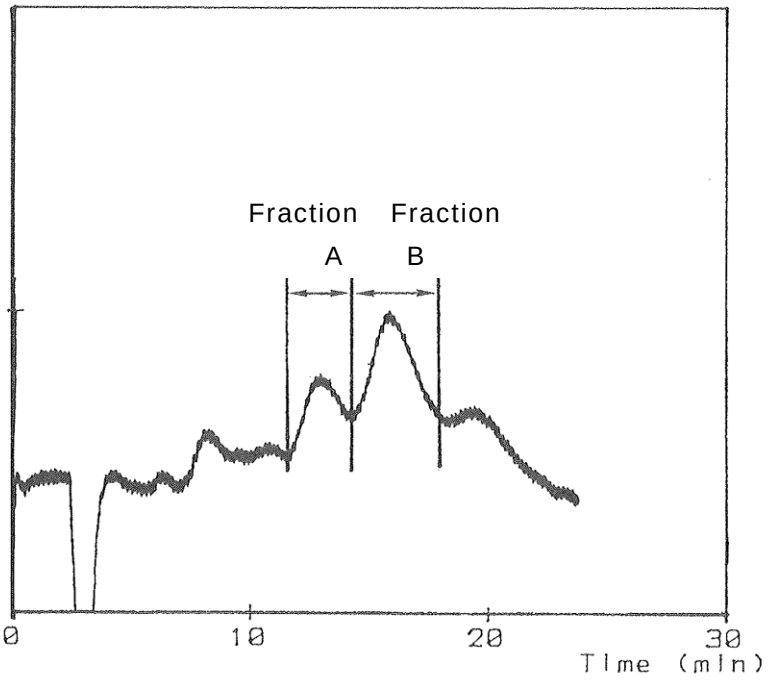
Pretest by HPLC

Column: 4.6x250mm HPLC column
Silica: NH SPS100-5SG
Mobile phase: Acetonitrile/H2O 65/35(w/w)
Flow rate: 2 ml/min
Detection: RI detector
Samples: 1. α–cyclodextrin
2. β–cyclodextrin
3. γ–cyclodextrin



(3-1) Preparative chromatography of cyclodextrin

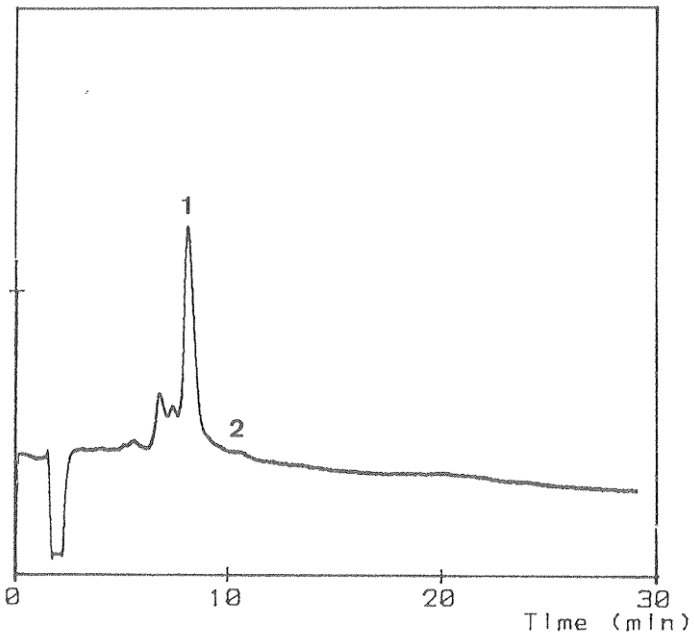
Column: 4.6x250mm HPLC column
Silica: NH SMB100-20/45SG
Mobile phase: Acetonitrile/H2O 65/35(w/w)
Flow rate: 20 ml/min
Detection: RI detector
Sample Loading: 100mg/1ml



(3-2) Analysis of fraction A Concentrated

Column: 4.6x250mm HPLC column
Silica: NH SPS100-5SG
Mobile phase: Acetonitrile/H2O 65/35(w/w)
Flow rate: 2 ml/min
Detection: RI detector

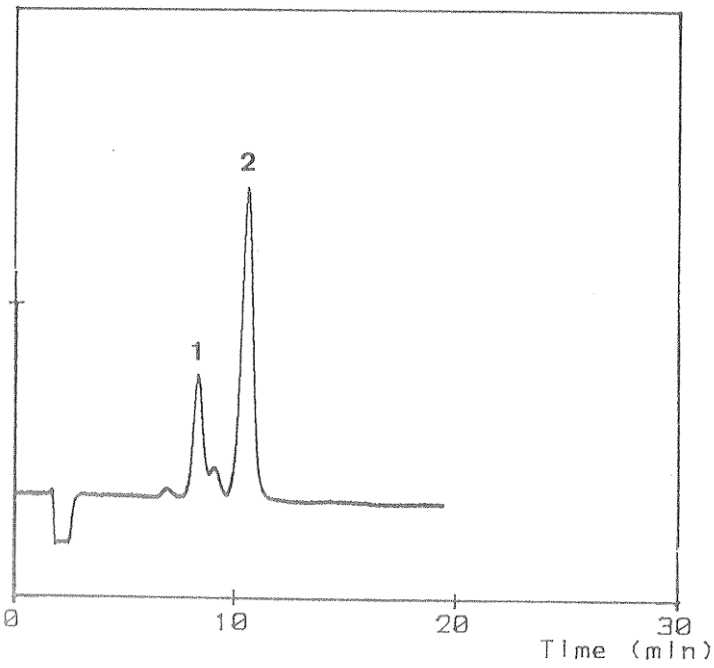
1. α–cyclodextrin
2. β–cyclodextrin



(3-3) Analysis of fraction B Concentrated

Column: 4.6x250mm HPLC column
Silica: NH SPS100-5SG
Mobile phase: Acetonitrile/H2O 65/35(w/w)
Flow rate: 2 ml/min
Detection: RI detector

1. α–cyclodextrin
2. β–cyclodextrin



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