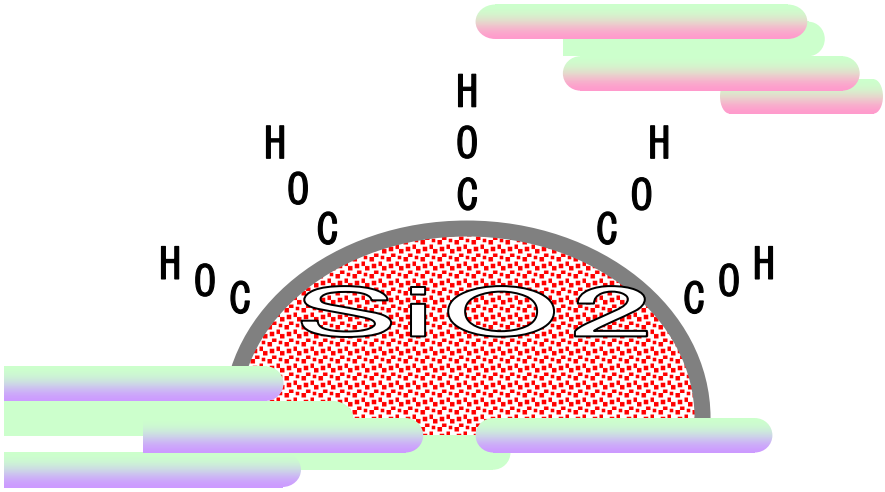
Technical Bulletin

CHROMATOREX

Diol Silica Gels

For Alternative to Amino Silica

Diol Silica



Introduction Diol Silica

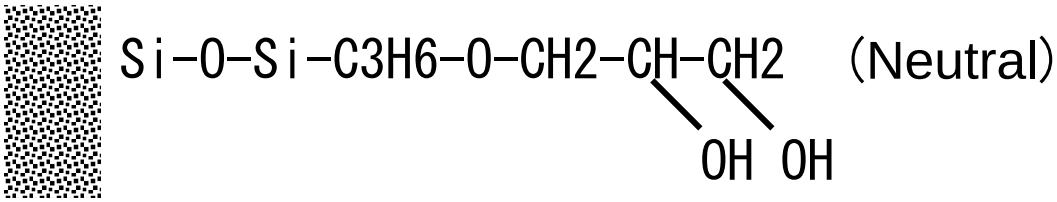
Surface modification of silica has been attempted to acquire unique adsorption property. Also, it has been developed a technique to introduce hydroxyl bond on silica surface; however, diol type silica was not widespread in chromatography field because a small advantage was found. Fuji Silysia Chemical Ltd. reviewed the production process and characteristics of Diol silica and we found out unique properties which ordinary silica doesn't have at normal phase chromatography. (Patent applied) This Diol silica is placed on the market for separation of problematic compounds so far.

Grade

4 kinds of different particle size grades are assorted based on 10nm pore size spherical silica. Please select from these grades as the application demands.

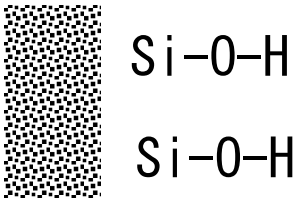
			(Application)
DIOL	**MB 100-75/200**	(A.P.S.110μm)	Open column
DIOL	**MB 100-40/75**	(A.P.S. 60μm)	Flash Column, Cartridge Column
DIOL	**SMB 100-20/45**	(A.P.S. 30μm)	Cartridge Column, HPLC
DIOL	**SMB 100-5**	(A.P.S. 5μm)	HPLC

【Surface characteristics of Diol silica】

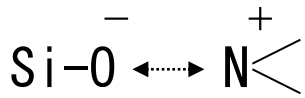


Alcoholic hydroxyl(C-OH) without ionicity exsist on silica surface. Mild hydrogen bond occur on the surface.

【Surface characteristics of silica gel】



(Acid)



Ionic adsorption between silanol bond and amine

Silanol bonds on silica surface are polarized and indicate high hydrogen-bonding. Also, acidic characteristics show ionic strong adsorption against basic compounds.

Characteristics of Diol Silica

Diol silica can be applied normal phase chromatography and has following unique properties compared with ordinary silica gel.

Silica gel :**Si-OH (Silanol group)** strong adsorption, minus charge

Diol silica :**C-OH (Alcoholic hydroxyl)** mild adsorption, neutral charge

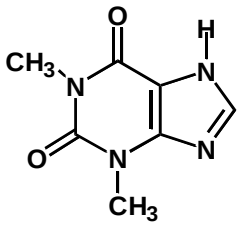
1. Same separation property as silica gel with weaker polar character at normal phase.
2. Specific strong adsorption property against phenolic compounds.
3. No strong adsorption property like a silica gel and superior separation property to basic compounds.

NH silica is effective to separate basic compounds in this field;however, it is unable to use under certain situation due to following problems.

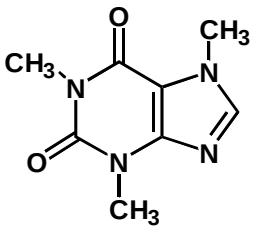
1. NH silica cannot be used separation for alkali-labile substances because its surface acts as strong base.
2. NH silica will react with ketones and aldehydes. It must be avoided for the application of compounds contain such groups and used for mobile phase such as acetone.
3. Acidic compounds will be adsorbed strongly on NH Silica. They cannot be eluted from the column bed.

Diol Silica can make up for these problems with unique character as a new separation method.

[Separation of Theophylline and Caffeine by Diol silica]



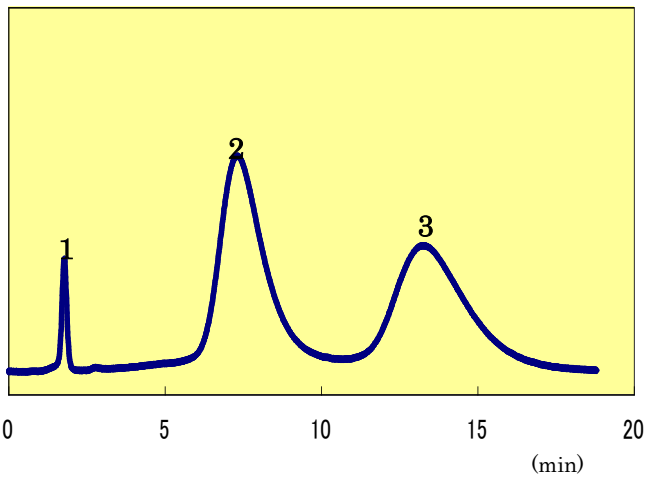
Theophylline



Caffeine

Samples

1. Benzene (t_0)
2. Theophylline
3. Caffeine



【Conditions】

Media	: DIOL MB 100-40/75
Column	: Cartridge Size 60 (28x100mm)
Mobile phase	: 10%EtOAc/n-hexane (w/w)
Flow rate	: 28ml/min
Detector	: UV254nm 0.32aufs

Separation of standard substances

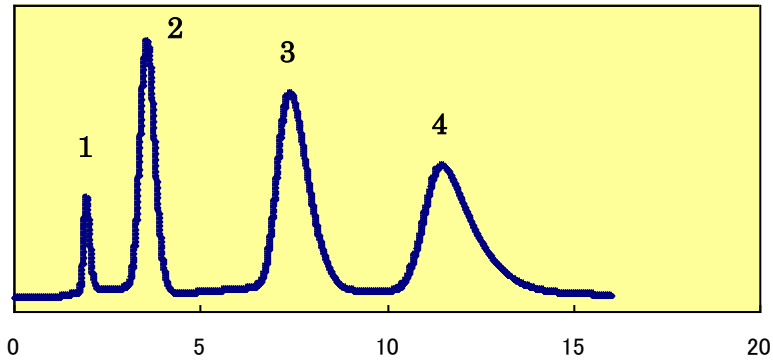
The figures below show the chromatograms of standard substances by Diol silica, silica gel and NH silica. The results of separation by Diol silica are excellent separation in spite of neutral, acidic, and basic compounds. On the other hands, pyridine of basic compound cannot be eluted by silica gel and, phenol of acid compound cannot be eluted by NH silica.

【Conditions】

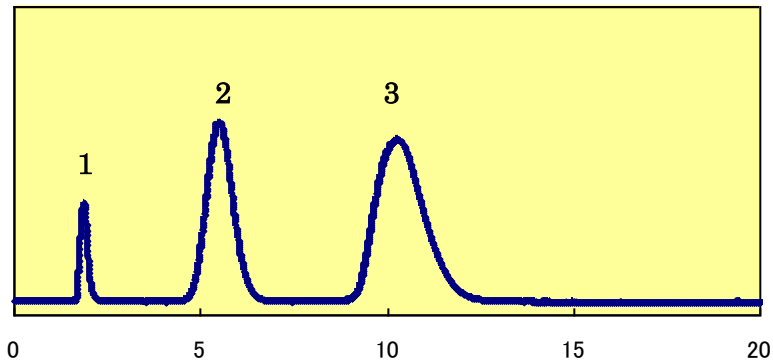
Column : Cartridge Size 60 (28x100mm)
Mobile phase : 10% ethyl acetate/n-hexane(w/w)
Flow rate : 28ml/min
Detector : UV254nm 0.32aufs

Samples

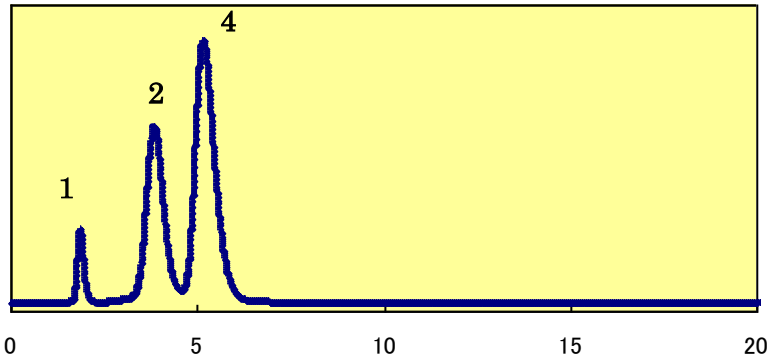
1 . Benzen (to)
2 . Dibutyl phthalate (neutral compound)
3 . Phenol (acidic compound)
4 . Pyridine (basic compound)



Packing media :
DIOL MB 100-40/75



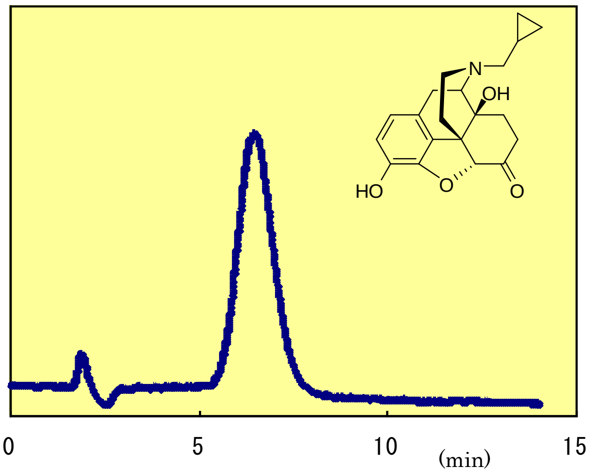
Packing media :
Silica gel MB 100-40/75



Packing media :
NH silica NH PSQ 100

Separation of alkaloids

1． Separation of Naltrexone



【Conditions】

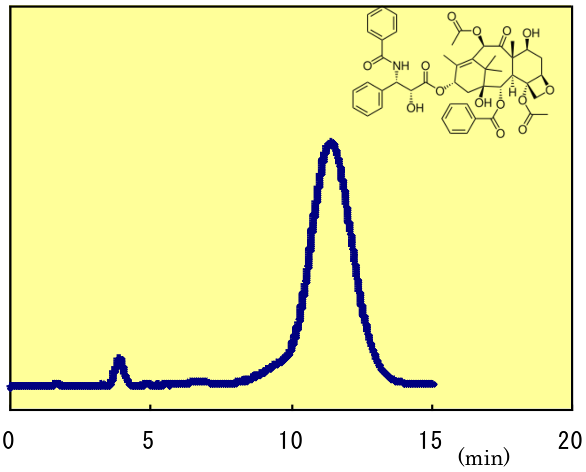
Packing media　　：DIOL　MB 100-40/75

Column：Cartridge: Size 60 (28x100mm)

Mobile phase：5% Isopropanol/n-hexane(w/w)

Flow rate　　：28ml/min

2． Separation of Taxol



【Conditions】

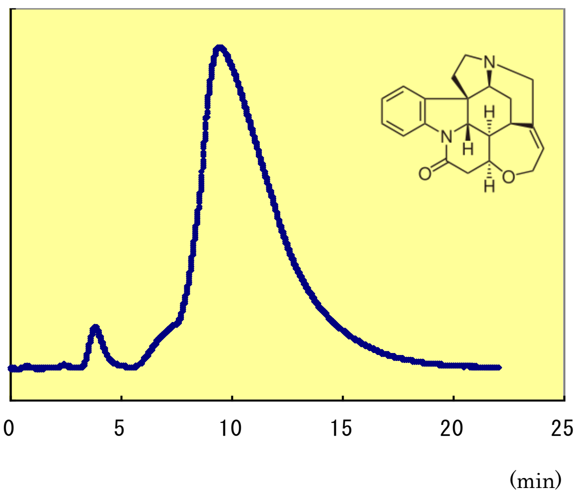
Packing media　　：DIOL　SMB 100-20/45

Column：Cartridge PRC M2　(20x250mm)

Mobile phase　　：Isopropanol/ n-hexane(4:6)

Flow rate　　：18ml/min

3． Separation of Strychnine



【Conditions】

Packing media　　：DIOL　SMB 100-20/45

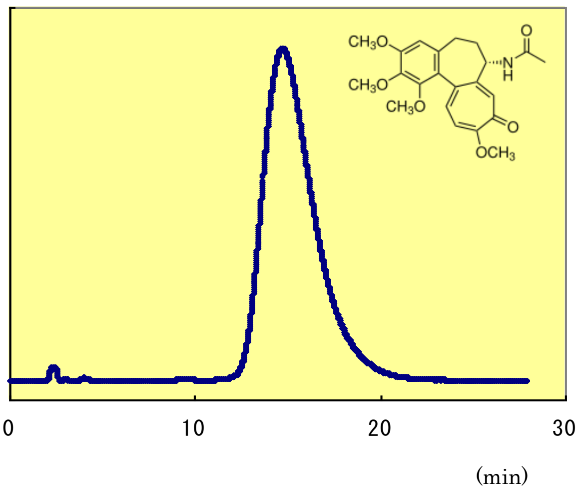
Column：Cartridge PRC M1　(10x150mm)

Mobile phase　　：Chloroform

Flow rate　　：18ml/min

Detector　　：UV254nm　0.64aufs

4． Separation of Colchicine



【Conditions】

Packing media　　：DIOL　SMB 100-20/45

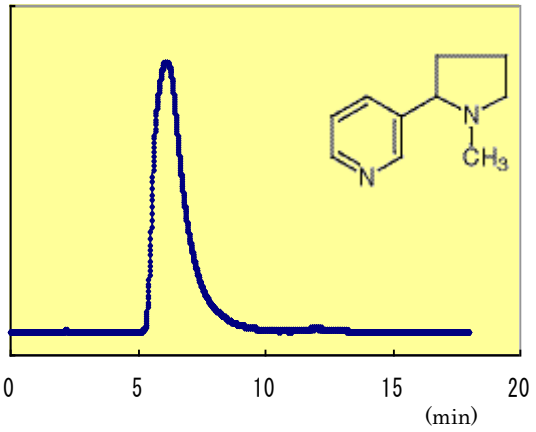
Column：Cartridge PRC M1　(10x150mm)

Mobile phase　　：Isopropanol/n-hexane (3:7)

Flow rate　　：18ml/min

Detector　　：UV242nm　0.64aufs

5. Separation of Nicotine



【Conditions】

Packing media : DIOL SMB 100-20/45

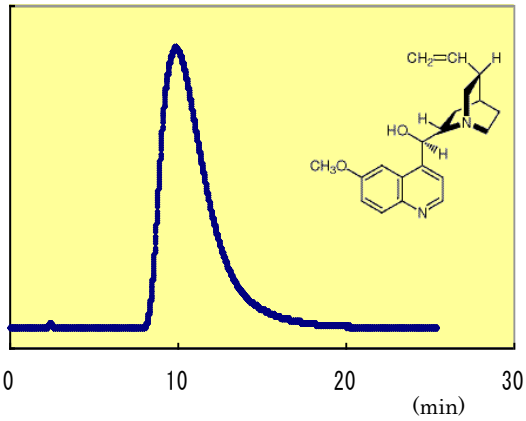
Column : Cartridge PRC M2 (20x250mm)

Mobile phase : Isopropanol/n- hexane (15:85)

Flow rate : 18ml/min

Detector : UV254nm 1.28aufs

6. Separation of Quinine



【Conditions】

Packing media : DIOL SMB 100-20/45

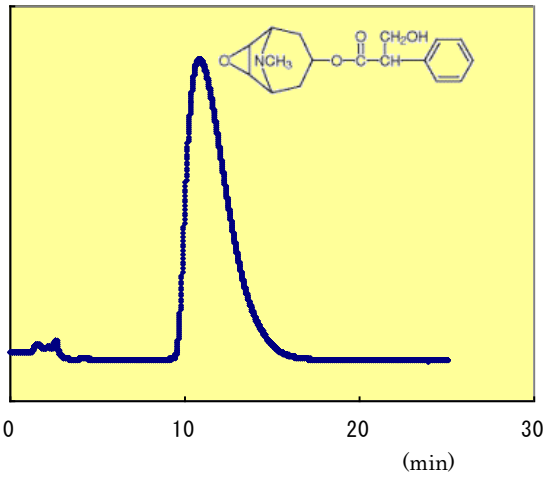
Column : Cartridge PRC M2 (20x250mm)

Mobile phase : Isopropanol/n-hexane(35:65)

Flow rate : 18ml/min

Detector : UV225nm 1.28aufs

7. Separation of Scopolamine



【Conditions】

Packing media : DIOL SMB 100-20/45

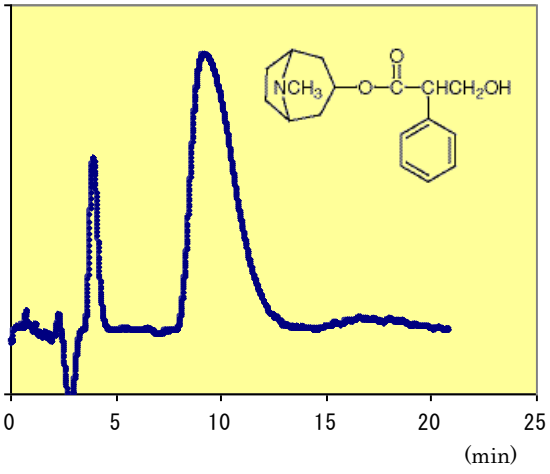
Column : Cartridge PRC M 1 (10x150mm)

Mobile phase : Isopropanol/n- hexane (2:8)

Flow rate : 18ml/min

Detector : UV210nm 0.64aufs

8. Separation of Atropine



【Conditions】

Packing media : DIOL SMB 100-20/45

Column : Cartridge PRC M 1 (10x150mm)

Mobile phase : Isopropanol/n- hexane (2:8)

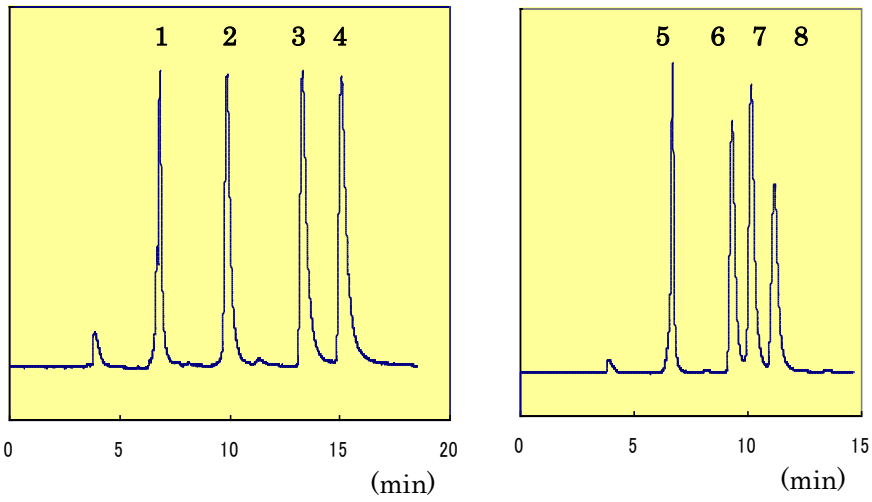
/3%triethylamine

Flow rate : 18ml/min

Detector : UV250nm 0.32aufs

Separation by HPLC

Separation of Naltrexone Derivatives



Provided samples from Prof. Nagase at KITASATO University.

【Conditions】

Packing media：DIOL SMB 100-5

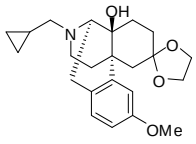
Column：4.6x250mm

Mobile phase : 2%EtOH/n-hexane (w/w)

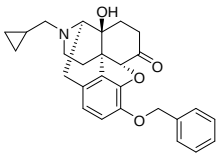
Flow rate：1ml/min

Detector : UV254nm 0.24aufs

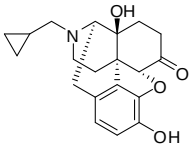
1.Morphinan2



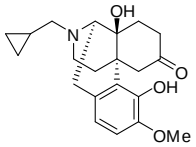
2.Benzyl ether



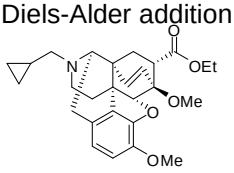
3. Naltrexone



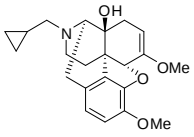
4. Morphinan1



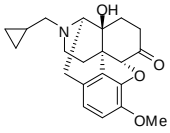
5.Diels-Alder addition



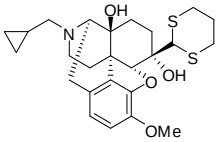
6.Enol ether



7.Methyl ether



8. Dithian addition



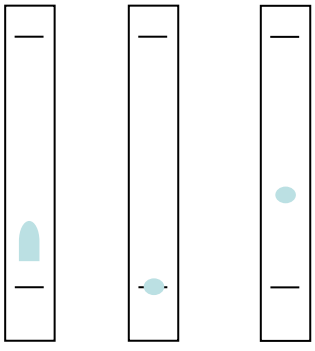
Diol TLC

We provide TLC plate corresponding to Diol silica for determination of analytical condition. The condition setting should be started with ethyl acetate/n-hexane or isopropanol /n-hexane as a standard. Please note that there are undetectable chemical compounds by UV and in the case of no molybdenum chromophore.

[Analysis examples]

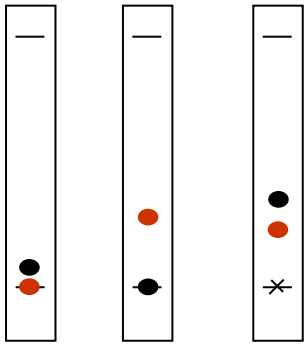
1. Separation of Naltrexone

Developing solvent: IPA/n-Hexane (2:8)



2. Separation of Caffeine , Theophylline

Developing solvent: IPA/n-Hexane (2:8)



Order Information

1. Bulk ACD Silica

Grades	100 g pack	1 kg pack	5 kg pack
DIOL MB100-75/200	-	Yes	Yes
DIOL MB100-40/75	-	Yes	Yes
DIOL SMB100-20/45	-	Yes	Yes
DIOL SPS100-5	Yes	Yes	-

2. TLC Plates ACD Silica

Grades	Dimension	Thickness	F Reagent	Plates/Pack
DIOL TLC Plates	20 x 20 cm	0.25 mm	F 254	10 pieces
DIOL TLC Plates	20 x 20 cm	0.25 mm	F 254	50 pieces

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