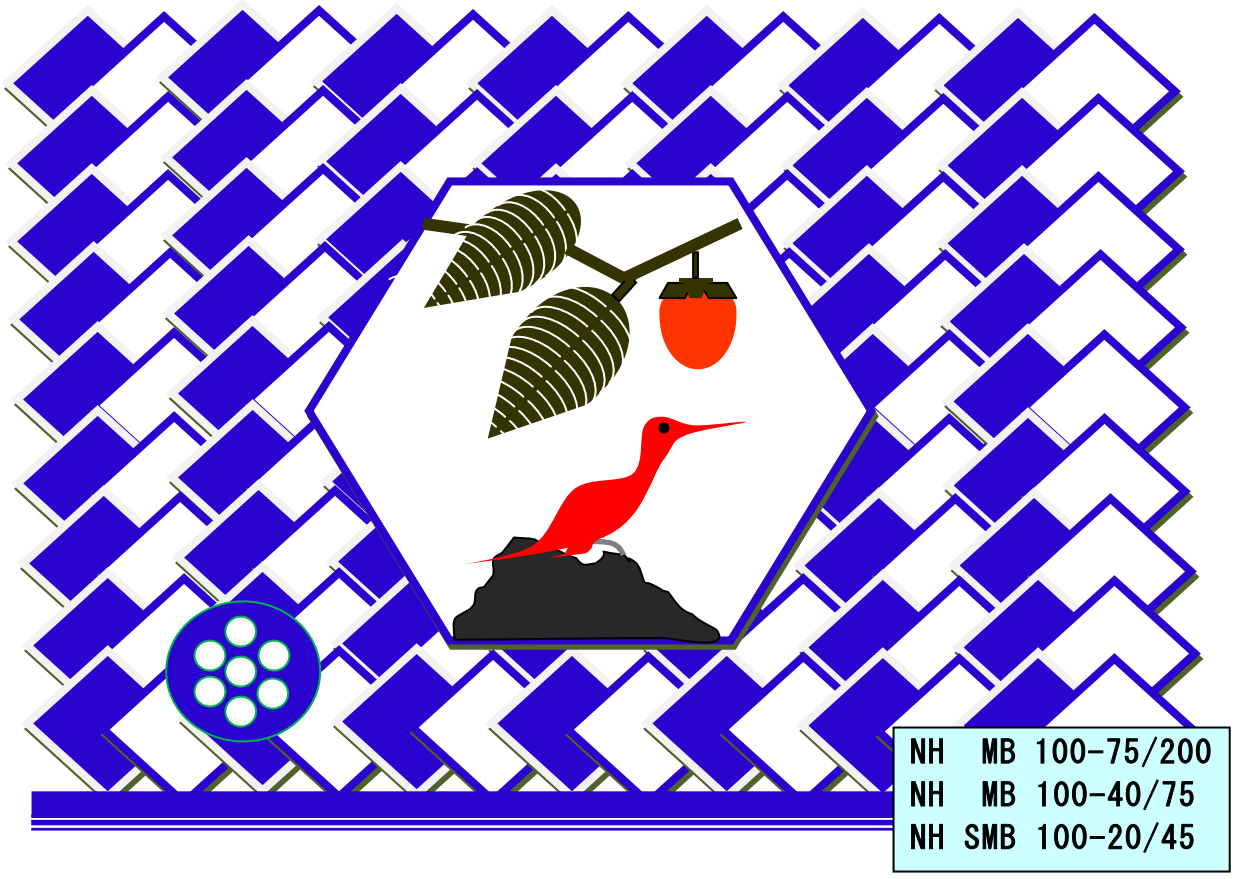
CHROMATOREX

Amino Silica Gel

A multi-purpose chromatography support



FUJI SILYSIA CHEMICAL LTD.

Introduction

LIQUID CHROMATOGRAPHY (LC) is a useful technique in purifying organic and natural compounds in laboratories and industrial production. Especially normal phase chromatography using silica gel and non-polar solvent is a popular choice because of high solubility of compounds and easy post-treatment. However, there is a difficulty to separate basic compounds such as alkaloids and nitrogen-containing compounds due to ionic interaction between silica gel and solutes. These alkaloids have superior efficacy in pharmaceutical fields both natural and synthesis products.

Fuji Silysia Chemical Ltd. (FSC), a manufacturer of large amounts of silica gel (CHROMATOREX) for LC since the last 40 years, has developed amino-propyl bonded silica, which can be used in various application techniques:

- Normal phase LC for basic compounds like alkaloids
- Reversed phase LC for vitamins or sugars application
- Metal scavenger removal/recovery for acid chlorides, isocyanates,..
- Precursor to develop chromatography chiral packings

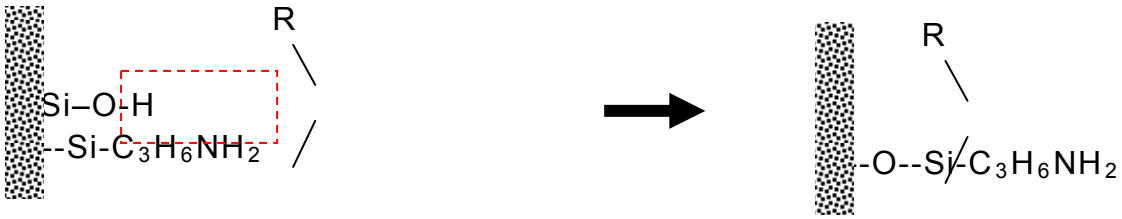
The CHROMATOREX amino-propyl bonded silica can be seen as a multi-purpose chromatography support.

Chromatorex Surface Characteristics

FSC introduces 3 major grades to cover a large field of applications:

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

Of course due to the availability of a large range of bare silica manufactured by FSC, FSC is able to provide additional product suitable for a particular application.
These grades resulted from silica surface treatment where the silanol groups react with the amino-propyl silane by chemical bonding:



Chromatorex Physical Properties

The table shows typical physical properties of NH silica gel. These grades are surface treated with amino silanes:

Items	NH SMB100-20/45	NH MB100-40/75	NH MB100-75/200
Surface area m^2/g	240	240	240
Pore volume ml/g	0.60	0.60	0.60
Bulk density g/ml	0.63	0.63	0.63
pH (5% slurry)	9.9	9.9	9.9
particle size μm	30	60	110

Separation of Neutral Compounds

The **NH Silica grade** shows similar chromatogram as normal phase silica, but shows lower adsorption power. K' value of NH grade is about half of corresponding silica gel.
Chromatography Operating Conditions:

- Column : 28 x 100 mm cartridge column
- Silica : **MB100-40/75** Vs **NH MB100-40/75**
- Mobile phase : Ethylacetate/n-hexane 10/90 (w/w)
- Flow rate: 28 ml/min
- Detection: UV-254 nm
- Samples : 1. Benzene
  2. Di-octyl phthalate
  3. Di-butyl phthalate
  4. Di-methyl phthalate

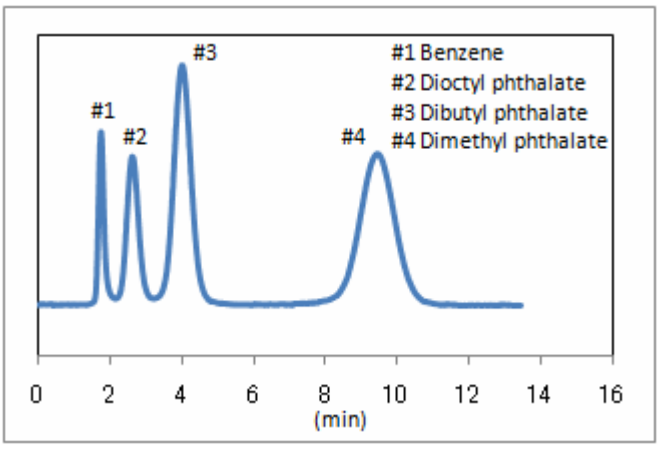


Fig.1 Chromatogram of **MB100-40/75**

Pressure 0.03MPa
k' DMP 4.39
N/m DMP 4333

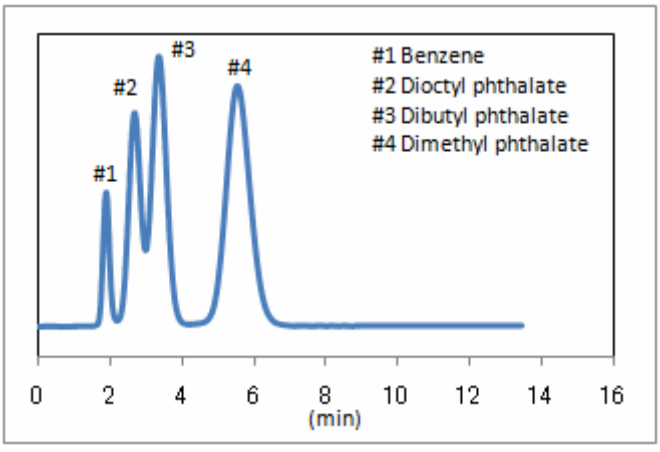
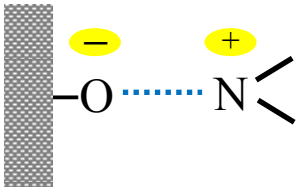


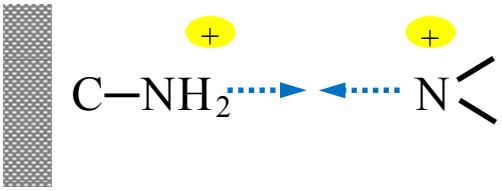
Fig.2 Chromatogram of **NH MB100-40/75**

Pressure 0.03MPa
k' DMP 2.13
N/m DMP 2715

Separation of Basic Compounds

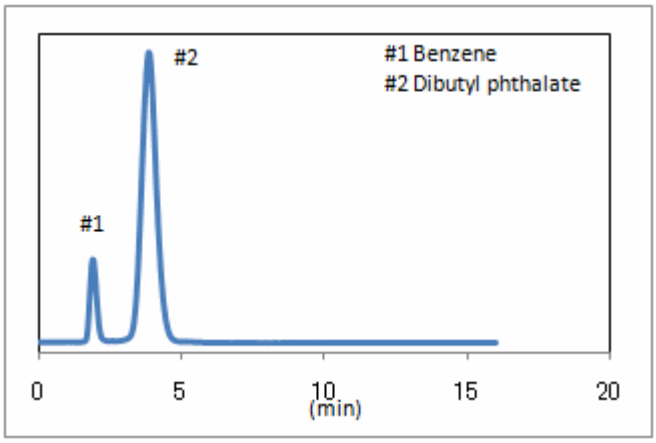
Some surface silanol groups act as a bronsted acid (silicic acid) and have characteristics to adsorb basic compounds by ionic bonds. NH silica grades are surface treated with amino groups to avoid the ionic interactions with the basic compounds. With basic compounds, the ionic bonds have stronger effects, adsorbing the basic compounds to the surface of silica gel. This causes the basic compounds to elute from the column with tailing and low yield. The surface of NH silica grades have plus charge that gives separation by only hydrogen interaction with the surface in the case of basic compounds.





Chromatography Operating Conditions:

- Column : 28 x 100 mm cartridge column
- Media : **MB100-40/75** Vs **NH MB100-40/75**
- Mobile phase : Ethylacetate/n-hexane 10/90 (w/w)
- Flow rate : 28 ml/min
- Detection : UV-254 nm
- Samples : 1. Benzene
  2. Di-butyl phthalate
  3. Nicotine



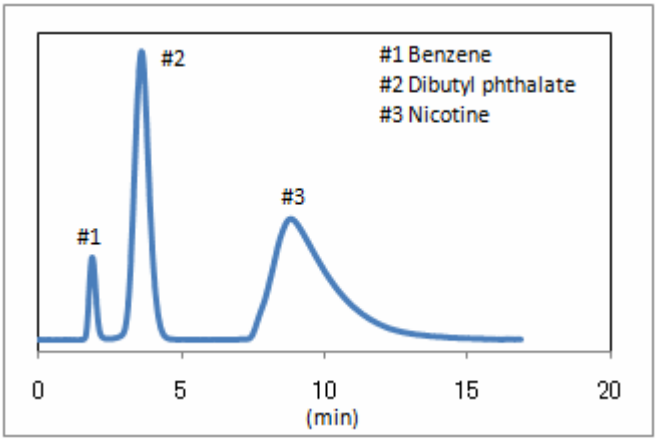
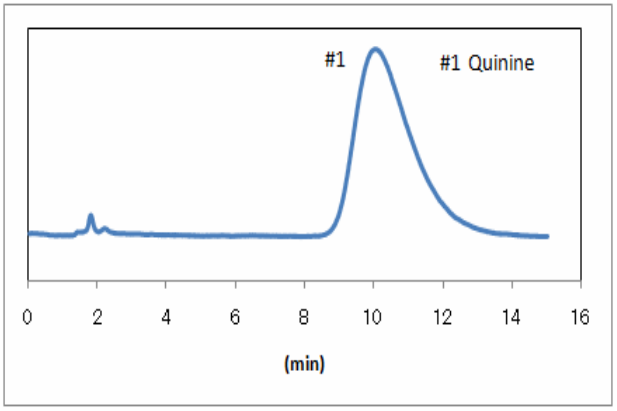


Fig. 3 Chromatogram of **MB100-40/75**

Fig. 4 Chromatogram of **NH MB100-40/75**

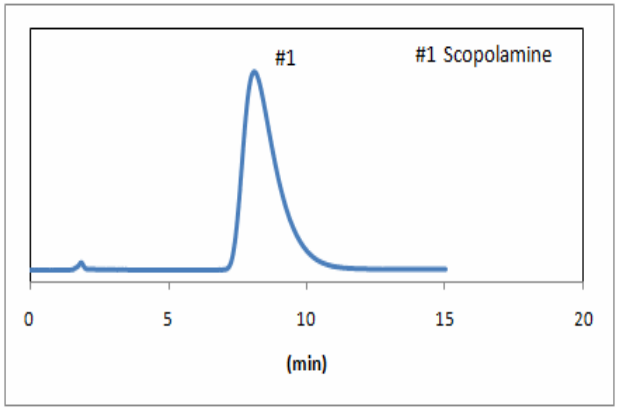
Other Applications

∗ Separation of Quinine



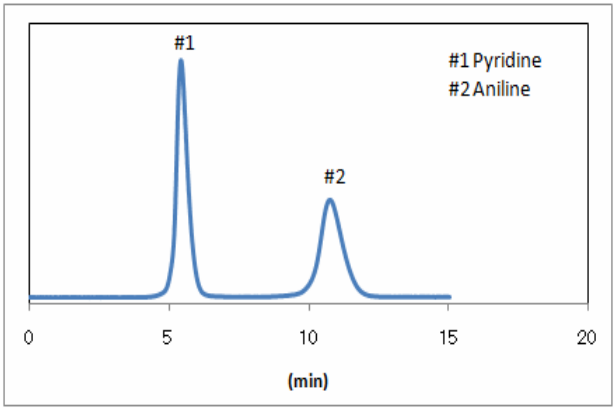
<Conditions>
Media: **NH SMB100-20/45**
Column : Cartridge Size60 (28x100mm)
Mobile phase : IPA/n-hexane (2/8)(w/w)
Flow rate: 28 ml/min
Detection: UV-235nm

∗ Separation of Scopolamine



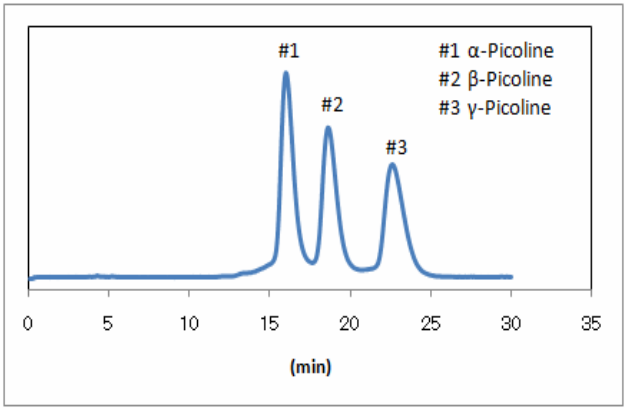
<Conditions>
Media: **NH SMB100-20/45**
Column : Cartridge Size60 (28x100mm)
Mobile phase : IPA/n-hexane (15/85)(w/w)
Flow rate: 28 ml/min
Detection: UV-210nm

∗ Separation of Pyridine and Aniline



<Conditions>
Media: **NH SMB100-20/45**
Column : Cartridge Size60 (28x100mm)
Mobile phase : Ethylacetate /n-hexane (1/9)(w/w)
Flow rate: 28 ml/min
Detection: UV-254nm

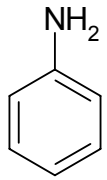
∗ Separation of Picolines



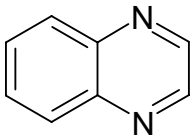
<Conditions>
Media: **NH SMB100-20/45**
Column : Glass column (20x250mm)
Mobile phase : Ethylacetate /n-hexane (5/95)(w/w)
Flow rate: 28 ml/min
Detection: UV-254nm

Separation of Amines on NH compared to NP and Diol

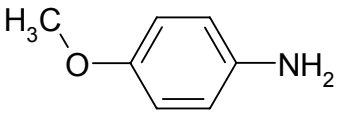
Three amines: Aniline, quinoxaline and P-Anisidine are separated on three different media to see the different properties and separation.



Aniline



Quinoxaline



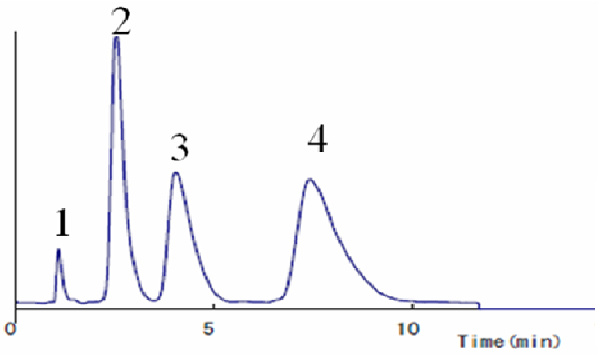
P-Anisidine

Chromatography Operating Conditions:

- Column : 20 x 60 mm cartridge column
- Flow rate: 16 ml/min
- Detection: UV-254 nm
- Samples : 1. Benzene
  2. Quinoxaline
  3. Aniline
  4. p-.Anisidine

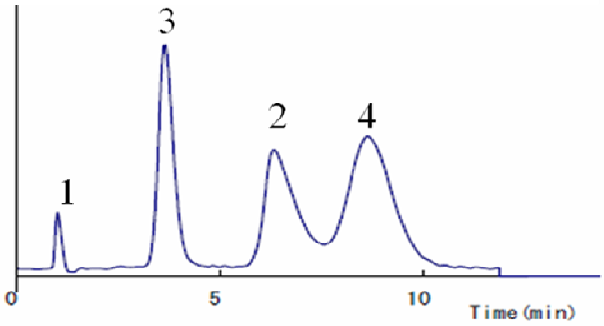
Media : **NH SMB100-20/45 (10g)**

Mobile phase : Ethylacetate/n-hexane 15/85 (w/w)



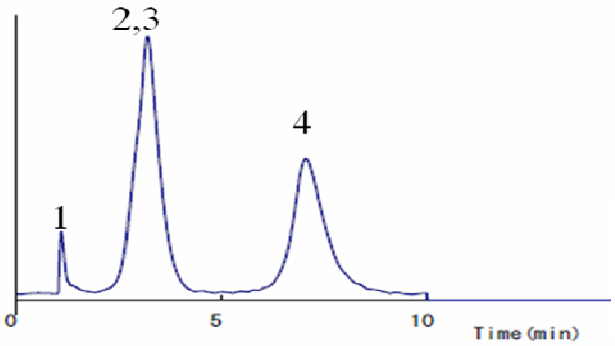
Media : **SMB100-20/45 (10g)**

Mobile phase : Ethylacetate/n-hexane 25/75 (w/w)



Media : **Diol SMB100-20/45 (10g)**

Mobile phase : Ethylacetate/n-hexane 10/90 (w/w)

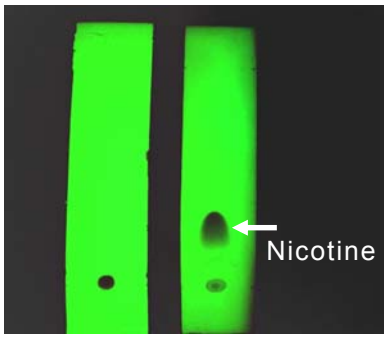


NHTLC

We provide TLC plate corresponding to NH 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.

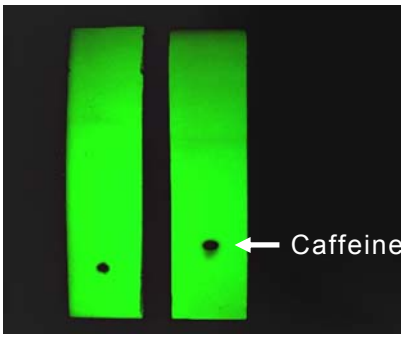
[Analysis examples]

1. Separation of Nicotine
Developing solvent:EtOAc/n-Hexane(1:9)



SI NH

2. Separation of Caffeine
Developing solvent: IPA/n-Hexane(2:8)



SI NH

Order Information

1. Bulk NH Silica

Grades	100g pack	500g pack	1 kg pack	8 kg pack
NH MB100-75/200	-	-	Yes	Yes
NH MB100-40/75	-	-	Yes	Yes
NH SMB100-20/45	-	Yes	Yes	Yes

2. TLC Plates NH Silica

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

3. Disposable Cartridges

Grades	DC Size	Dia. x L	DC/Box	Box/Carton
NH MB100-40/75	60	28 x 100 mm	20	8
NH SMB100-20/45	60	28 x 100 mm	20	8

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