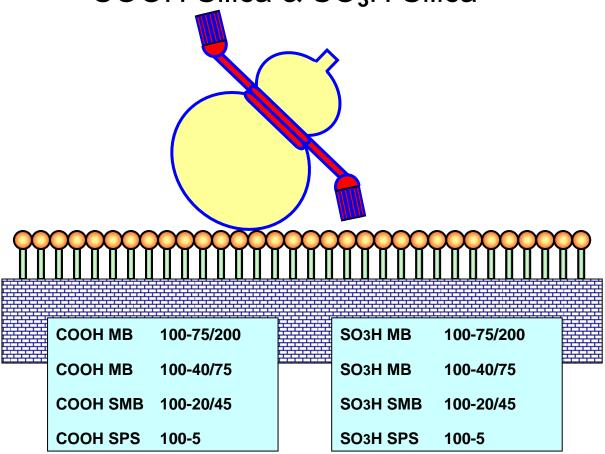
CHROMATOREX ACD Silica Gels

For the separation of acid compounds

COOH Silica & SO₃H Silica



Introduction

Liquid chromatography is a widely used technique for purification of organic compounds. Normal phase chromatography using silica gel and non-polar solvents such as hexane and ethyl acetate is a popular choice because of a high solubility of compounds and easy post-treatment.

However, compounds with basic or acidic characteristics are often difficult to separate using bare silica gel. Though AMINO and DIOL media have been developed for compounds that have basic separation difficulty, there is not yet a proper media to separate acid compounds with carboxyl group, for example.

Fuji Silysia Chemical Ltd. developed an appropriate separation, Chromatorex ACD Silica, for acidic compounds by introducing COOH and SO₃H bonds on the silica surface (patent applied).

Normal Phase Bare & Bonded Silica grades

The ACD Silica grades complete the normal phase bare and bonded silica of Fuji Silysia Chemical.

For the separation of organic compounds by normal phase liquid chromatography, it is necessary to select the proper media based on its characteristics:

- Neutral compounds are well separated by bare silica gel.
- NH silica and DIOL silica are used for basic compounds separation.
- Now acid compounds can be separated with our new ACD Silica.



Surface characteristics of Chromatorex ACD silica

Si-
$$C_3H_6$$
-NH-CO- CH_2 - CH_2 -COOH (COOH Silica)
Si- C_3H_6 -O- CH_2 - CH - CH_2 -SO $_3$ H (SO $_3$ H Silica)
OH

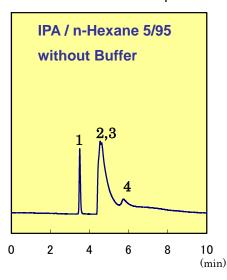
Advantages of ACD Silica

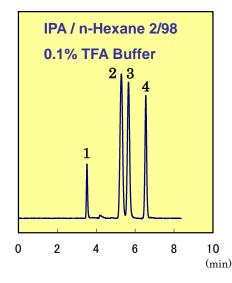
In solution, a part of acidic organic compounds with carboxylic acid are dissociated:

R-COOH \leftarrow R-COO⁻ + H⁺

ACID COMPOUNDS SEPARATION BY NORMAL PHASE: SPS100-5

To separate acidic compound by normal phase bare silica gel, the addition of acid is necessary in the solution. It is no problem to use pH buffering agent in the case of analytical separation; however when performing preparative separations, the pH buffering agent must be removed later in the process.





Operating ConditionsMedia:SPS 100-5Column:4.6 x 150 mmFlow rate:1ml / min

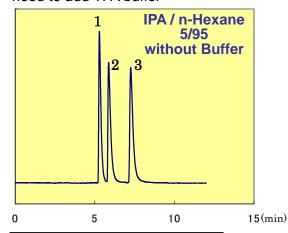
Detector: UV 254 nm, 0.32 aufs

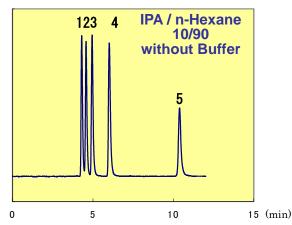
Samples

- Benzene (t₀)
 o-Toluic acid
- Benzoic acid
- 4. trans-Cinnamic acid

ACID COMPOUNDS SEPARATION BY ACD SILICA: COOH SPS100-5 & SO3H SPS100-5

ACD silica with an "immobilized acid functional group" works as well as buffer, therefore no need to add TFA buffer





Operating Conditions

Media: COOH SPS 100-5

Column: 4.6 x 250 mm

Flow rate: 1ml / min

Detector: UV 254 nm, 0.32 aufs

Samples

- 1. o-Toluic acid
- 2. Benzoic acid
- 3. trans-Cinnamic acid

Operating Conditions

Media: **SO3H SPS 100-5** Column: 4.6 x 250 mm

Flow rate: 1ml / min

Detector: UV 254 nm, 0.32 aufs

Samples

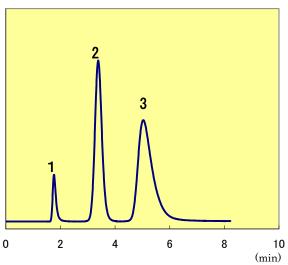
- 1. o-Toluic acid 4. Salicylic acid
- 2. Benzoic acid 5. Phthalic acid
- 3. trans-Cinnamic acid

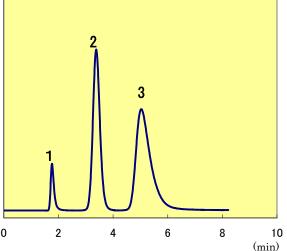
-2-

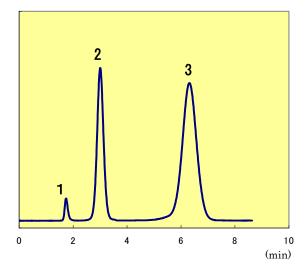
Standard Separation using Disposable Cartridges

To ease the evaluation of a separation disposable cartridges are available in a convenient size (60 ml, or 28 x 100 mm) before scaling up in large bulk quantity.









Operating Conditions

COOH SMB100-20/45 Media: Column: Disposable Cartridge Size 60

28 x 100 mm

Mobile Phase: IPA / n-Hexane (10/90)

Flow rate: 28 ml / min

Detector: UV 254 nm, 0.32 aufs

Samples

1. Benzene (t₀)

2. Di-Methyl Phthalate

trans-Cinnamic acid

Operating Conditions

Media: SO3H SMB100-20/45

Column: Disposable Cartridge Size 60

28 x 100 mm

Mobile Phase: IPA / n-Hexane (10/90)

Flow rate: 28 ml / min

UV 254 nm, 0.32 aufs Detector:

Samples

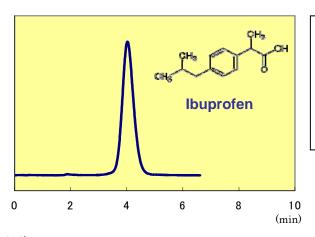
Benzene (t₀)

Di-Methyl Phthalate

Phthalic acid

Separation of drugs using Disposable Cartridges

1. Ibuprofen



Operating Conditions

Media: COOH SMB100-20/45
Column: Disposable Cartridge

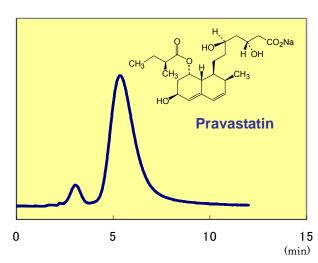
Size 60 (28 x 100 mm)

Mobile Phase: IPA / n-Hexane (5/95)

Flow rate: 28 ml / min

Detector: UV 254 nm, 0.32 aufs

2. Pravastatin



Operating Conditions

Media: COOH SMB100-20/45
Column: Disposable Cartridge

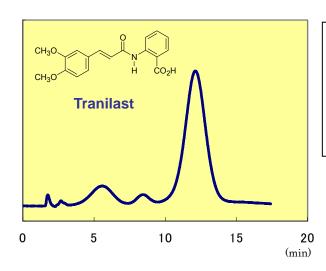
Size 60 (28 x 100 mm)

Mobile Phase: IPA / n-Hexane (40/60)

Flow rate: 28 ml / min

Detector: UV 254 nm, 0.32 aufs

3. Tranilast



Operating Conditions

Mobile Phase:

Media: SO₃H SMB100-20/45
Column: Disposable Cartridge

Size 60 (28 x 100 mm) IPA / n-Hexane (10/90)

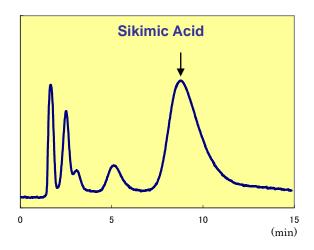
Flow rate: 28 ml / min

Detector: UV 254 nm, 0.32 aufs

Separation of Natural Products using Disposable Cartridges

1. Separation of Sikimic acid from Star Anise

→Dissolve, Filtration → Extract sample Sikimic Acid



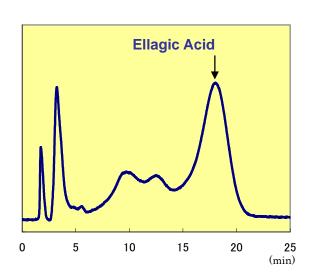
Operating Conditions

Media: COOH SMB100-20/45
Column: Disposable Cartridge
Size 60 (28 x 100 mm)
Mobile Phase: IPA / n-Hexane (40/60)

Flow rate: 28 ml / min

Detector: UV 254 nm, 0.32 aufs

2. Separation of Ellagic acid from Pomegranate



Operating Conditions

Media: SO₃H SMB100-20/45
Column: Disposable Cartridge
Size 60 (28 x 100 mm)

Size 60 (28 x 100 mm) IPA / n-Hexane (10/90)

Mobile Phase: IPA / n-Hex Flow rate: 28 ml / min

Detector: UV 254 nm, 0.32 aufs

ACD Silica on TLC Plates

In addition to bulk and disposable cartridges, FSC provides TLC plates corresponding to ACD silica (COOH silica and SO₃H silica) for determination of analytical conditions. 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.

Example 1:

Comparison between COOH TLC and Silica Gel TLC

Developing solvent:

IPA / n-Hexane (10/90)

Samples

- 1. o-Toluic acid
- 2. Benzoic acid
- 3. trans-Cinnamic acid



Example 2:

HPLC separation by SO₃H silica & TLC comparison between SO₃H, COOH and Silica

Developing solvent:

IPA / n-Hexane (10/90)

Operating Conditions:

Media: **SO3H SPS 100-5** Column: 4.6 x 250 mm

Mobile Phase: IPA / n-Hexane (10/90)

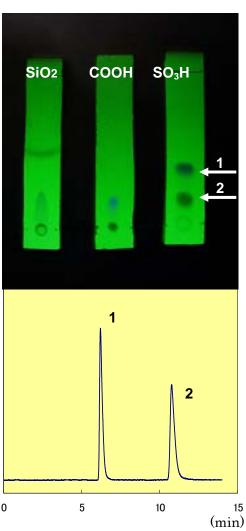
Flow rate: 1ml / min

Detector: UV 254 nm, 0.32 aufs

Samples

1. Salicylic acid

2. Phthálic acid



Order Information

1. Bulk ACD Silica

Grades	100 g pack	1 kg pack	5 kg pack
COOH MB100-75/200	-	Yes	Yes
COOH MB100-40/75	-	Yes	Yes
COOH SMB100-20/45	-	Yes	Yes
COOH SPS100-5	Yes	Yes	-
SO ₃ H MB100-75/200	-	Yes	Yes
SO ₃ H MB100-40/75	-	Yes	Yes
SO ₃ H SMB100-20/45	-	Yes	Yes
SO ₃ H SPS100-5	Yes	Yes	-

2. TLC Plates ACD Silica

Grades	Dimension	Thickness	F Reagent	Plates/Pack
COOH TLC Plates	20 x 20 cm	0.25 mm	F 254	10 pieces
SO ₃ H 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
COOH MB100-40/75	60	28 x 100 mm	20	8
COOH SMB100-20/45	60	28 x 100 mm	20	8
SO ₃ H MB100-40/75	60	28 x 100 mm	20	8
SO ₃ H SMB100-20/45	60	28 x 100 mm	20	8

FUJI SILYSIA CHEMICAL LTD.

2-1846 Kozoji-cho,

Kasugai-shi, Aichi-ken,

Japan 487-0013

Phone : +81 568 51 2516

Fax : +81 568 51 8557

Website: http://www.fuji-silysia.co.jp

E-mail: chromato-jpn@fuji-silysia.co.jp

FUJI SILYSIA CHEMICAL SA

International Chromatography Center

En Budron E 9

CH-1052 Le Mont-sur-Lausanne Switzerland

Phone : +41 21 652 3436 Fax : +41 21 652 4737

E-mail: Fuji.Silysia.SA@fuji-silysia.co.jp

Sep. 2009