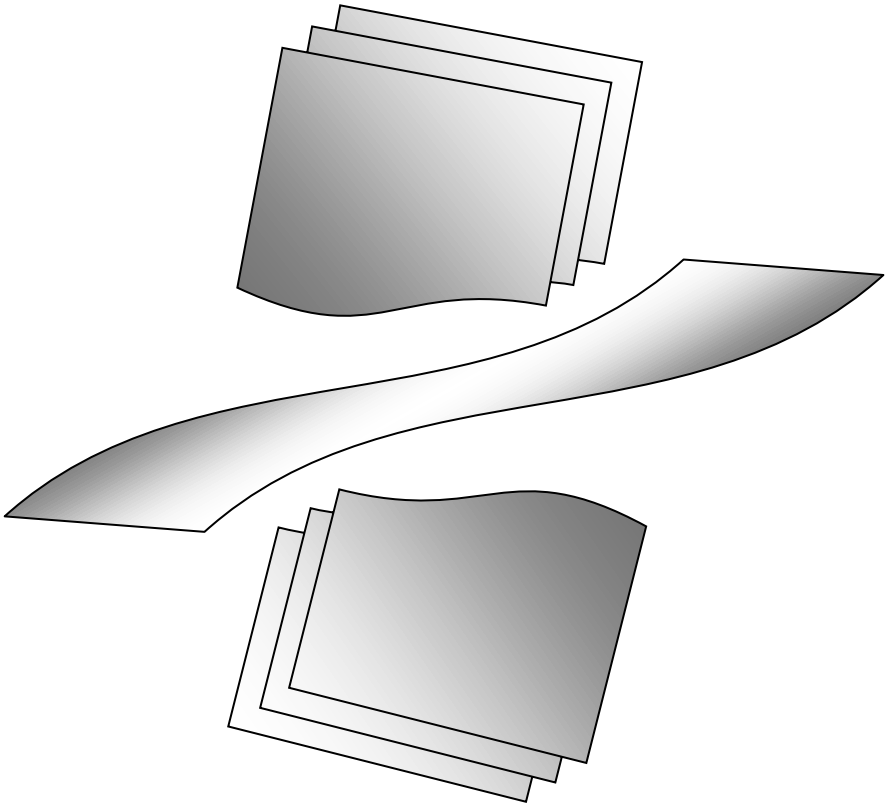
Technical Bulletin

# CHROMATOREX

# Bare Silica Gels

*For Flash and Industrial Chromatography*

## GS60 Silica



FUJI SILYSIA CHEMICAL LTD

## *<GS SILICA GEL>*

GS Silica gel is for column chromatography by FUJI SILYSIA Chemical LTD.
GS silica gel is granular in shape with 6 nm pore size. GS silica gel is available in three kinds of particle size grade to serve any applications from laboratory to industrial separation.

Our GS60-20/45, GS60-40/75 and GS60-75/200 are suitable for a wide range of flash chromatography needs. The high purity and sharp particle size distribution of this silica perform to the strictest requirements. We believe that GS grade will assist you with both your laboratory and production requirement.

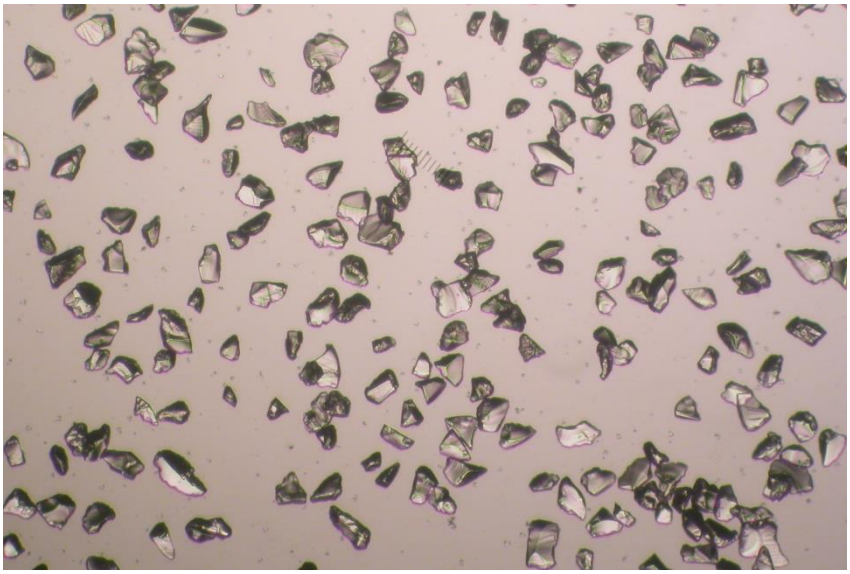
## *<PHYSICAL PROPERTIES>*

The table below shows typical properties physical of GS60-40/75 silica gel. The values of surface area pore volume show that GS silica gel is a typical 6nm grade silica gel. The pH and conductivity values that are measured in 5 % slurry show that GS silica gel is a highly pure substance. Since GS silica gel has similar physical properties for a range of particle sizes, the GS silica grades can easily be changed from laboratory to production scale.

Physical Properties of GS silica gel

| Items | Typical measured values |
|---|---|
| Surface Area ($m^2/g$) | 490 |
| Pore Volume (ml/g) | 0.72 |
| pH | 7.2 |
| Conductivity ($\mu$ s) | 20 |
| Bulk density (ml/g) | 0.5 |
| Water content (wt%) | 5.0 |

## *<MICROSCOPE PICTURE>*



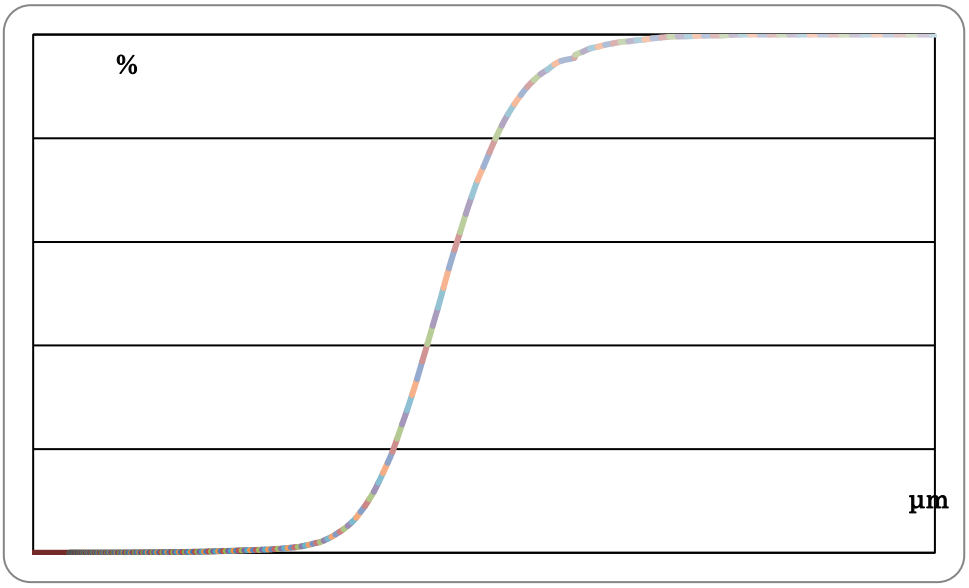
100 $\mu$ m

## *<PARTICLE SIZE DISTRIBUTION>*

The table and graph below display the particle size distributions of GS60-40/75. Particle size distributions are showed as $d_{90}/d_{10}$ values. These derived values represent the sharpness of peak on the cumulative particle distribution curve. When the value is closer to 1, the distribution is sharper.

| | Ave. particle size ( $\mu$ m) | Distribution(d90/d10) |
|---|---|---|
| GS60-40/75 | 60 | 1.62 |

**Accumulated particle size distribution chart**



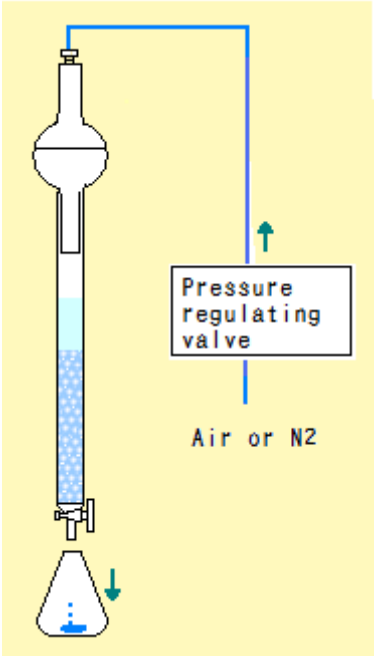
## *<FLASH CHROMATOGRAPHY>*

The name and technique for "flash chromatography" were proposed by Still et.al in 1978.[1] Flash chromatography has become a popular procedure for preparative separation in the field of organic chemistry. It is mainly utilized in lab scale preparative separations. The method is simple and easily performed in a short amount of time. Because of this, it is widely used as an alternative to open column chromatography and preparative TLC. GS60-40/75 performs very well in flash chromatography. This section explains the system and operations of flash chromatography.

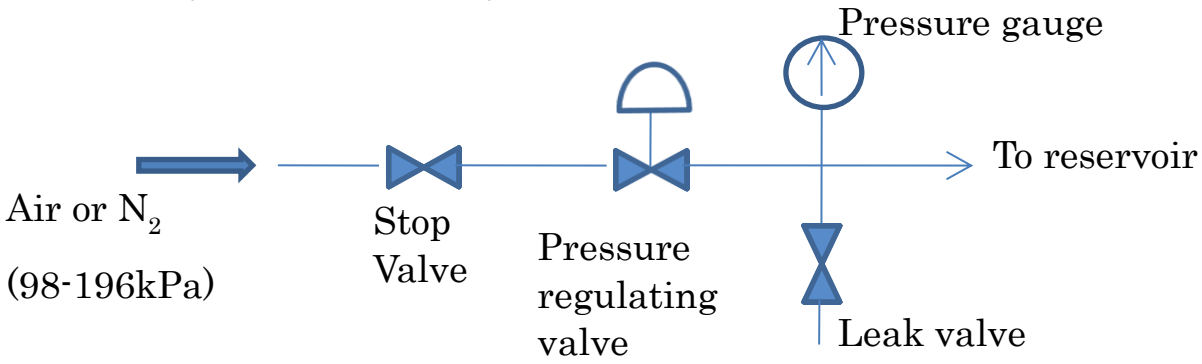
--Flash Chromatography System--

Flash chromatography is done using a glass column connected with compressed air with regulator line filled with air at constant pressure to elute the solvent in a short time (during 30 min.).

The figure to the right shows a typically recommended system for a lab. The flow rate is recommended to be 5cm/min (linear velocity) and is controlled by the pressure of air. The volume of the reservoir is selected by column diameter and separation time. It is convenient to have the outlet of the column connected to the detector to monitor separation condition. Otherwise, a fraction collector is used to collect eluents from the column.




### Pressure Adjustment Valve System




1) Journal of Organic Chemistry 43 (14) 2923 (1978)

Silica gel of 30-50 μm particle size is normally used in flash chromatography. Whereas, 100 μm is used for open column chromatography.

**GS silica gel for flash chromatography ・・・GS60-20/45, GS60-40/75**

**GS silica gel for open column ・・・GS60-75/200**

Basically, flash chromatography is a kind of open column chromatography that uses fine silica gel under pressurized air. It shows a higher performance in several areas more than normal open column in a shorter period of time.

## *<PACK AND SEPARATION PROCEDURE>*

The following is an example of flash chromatography operation with a 2 cm I.D. glass column. We recommend the slurry packing process.

1. Plug the end of the column with cotton.
2. Place about a 3mm depth of glass beads on top of the plug.*
3. Weigh 25g of silica gel in 200ml beaker.
4. Pour about 200ml of solvent into beaker and stir to make a slurry.
5. Clamp column vertically, then set 100ml reservoir at the top of column. Put funnel on the reservoir.
6. Pour solvent into column about 10 cm height. Tap the side of column until the glass beads lay flat.
7. Open the stop cock, and pour silica slurry into the column.
8. Connect the top of the reservoir to the pressurizing system. Next, pressurize the column to 80-100 kPa.
9. Wash the column wall with solvent, then place another layer of glass beads 3mm thickness on top of the silica.
10. Adjust the flow rate about 5cm/min by controlling the pressure.
11. Adjust the position of the solvent surface to the top of the silica layer.
12. Connect the outlet of the column to the detector with Teflon tubing.
13. Carefully pour the sample solution to the top of the silica layer.
14. Open the stop cock to adsorb sample into the silica. Wash the column wall and glass beads with small amount of solvent. Open the stop cock to adsorb the sample again.
15. Add solvent carefully to the top of the column. Connect the 500ml reservoir to the column.
16. Pour about 300ml of solvent into the reservoir.
17. Adjust the pressure to the preliminarily tested value.
18. Open the stop cock to start the chromatography process.

* Sea sand can also be used

## *<COMPARISON TO OTHER BRANDS>*

The following data shows a comparison of physical and chemical properties between GS60-40/75 and another commercially available silica gel for flash chromatography. Brand A have low surface areas compared to GS60-40/75. Brand A had high conductivity in water slurry because of high water soluble impurities. This high impurity was also displayed in chemical analysis. These impurities can sometimes elute from the column containing the purified product, especially if the solvent is water or a high methanol system.

The GS60-40/75 has a sharp particle size distribution compared with brand A. Generally, silica that has a broad particles size distribution may cause high pressure in the column due to fine particles and low separation performance due to coarse particles. GS60 40/75 shows a good particle size distribution and consequently it achieves superior performance over the other brands in flash chromatography at low pressure.

[Physical properties]

| | GS60 40/75 | Brand A |
|---|---|---|
| Surface area ($m^2/g$) | 492 | 435 |
| Pore volume (ml/g) | 0.72 | 0.73 |
| Ave. Pore Diameter(nm) | 5.9 | 6.7 |
| pH | 7.2 | 6.8 |
| Conductivity ($\mu$ s) | 14.4 | 244 |
| Bulk density (ml/g) | 0.53 | 0.52 |
| Water content (wt%) | 5.4 | 6.5 |
| Ave. Particle size ($\mu$ m) | 59 | 52 |
| Distribution (d90/d10) | 1.62 | 1.82 |

[Chemical properties] (Impurities)

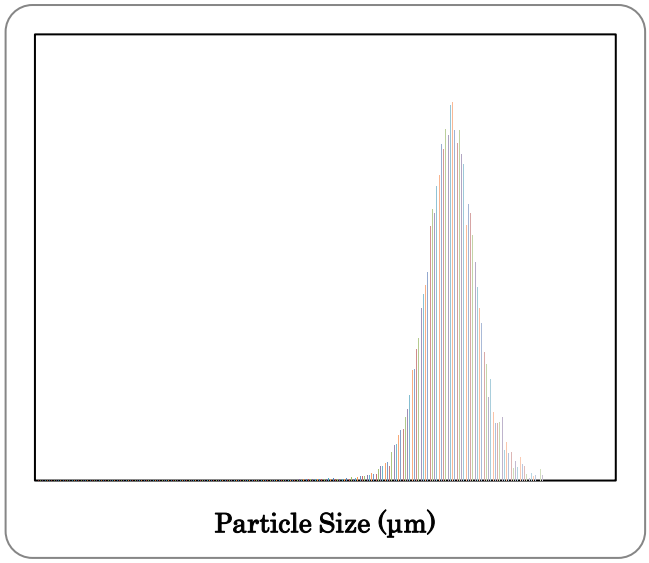
| | | |
|---|---|---|
| Na ppm | 110 | 990 |
| Ca ppm | 170 | 840 |
| Fe ppm | 30 | 80 |
| Al ppm | 20 | 180 |

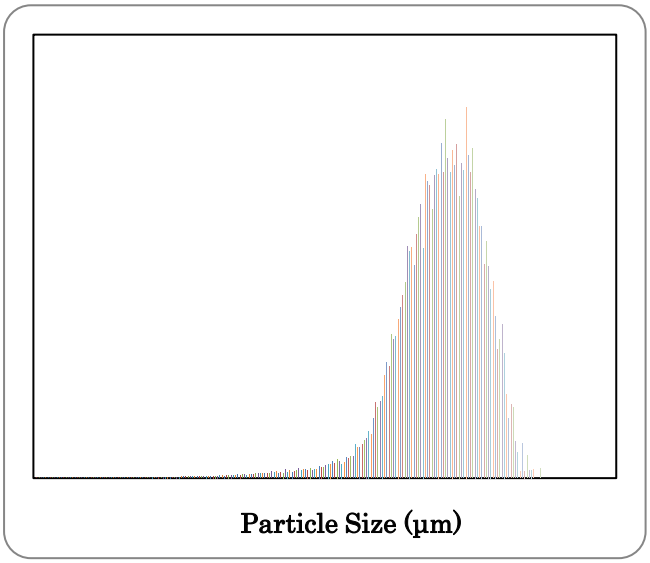
[Chromatographic properties]

| | | |
|---|---|---|
| Pressure (kPa) | 13 | 13 |
| Column height (mm) | 170 | 179 |
| Capacity factor ($k'_{DMP}$) | 6.11 | 5.12 |
| Number of plate ($N/m_{DMP}$) | 3800 | 3400 |

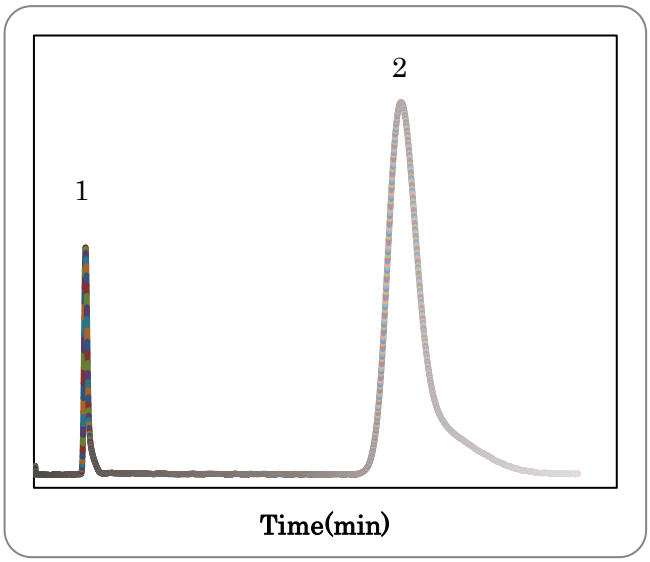
GS60-40/75 Brand A

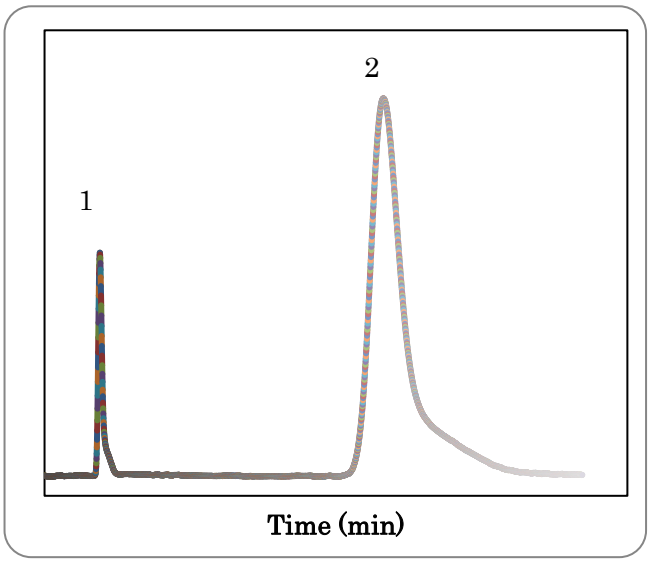
[Particle Size Distribution Chart]




[FlashChromatogram]



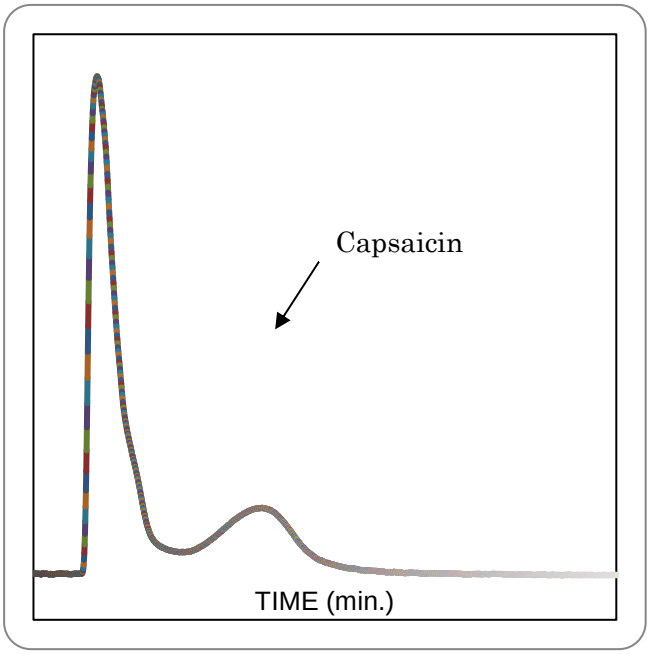




1. Benzene 2. Dimethylphthalate

## *<SEPARATION EXAMPLES>*

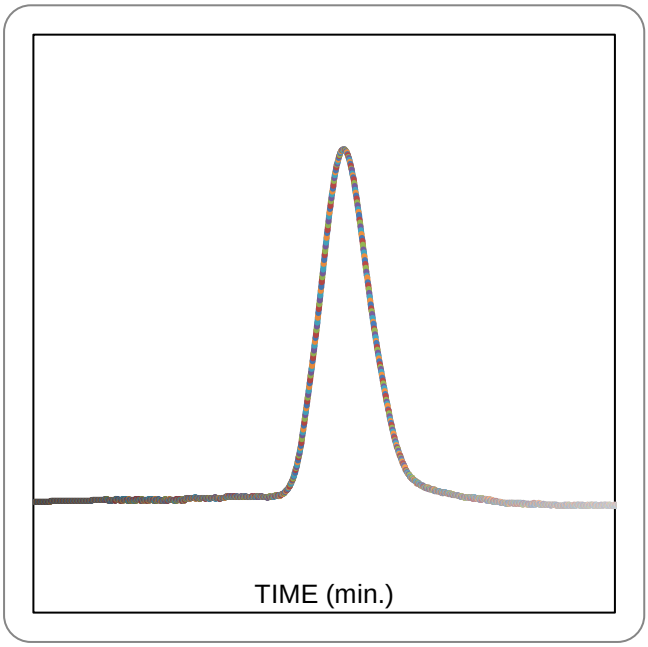
**Separation of red pepper**

Column : 20mm I.D. Glass column
Media : GS 60-40/75(20g)
Column height: 135mm
Pressure : 10 kPa
Mobile phase : 15%EtOH/n-Hexane (w/w)
Flow rate : 5cm/min. (15.7ml/min)
Detector : UV280nm




**Separation of Coenzyme Q-10**

Column : 20mm I.D. Glass column
Media : GS 60-40/75(20g)
Column height: 132mm
Pressure : 8 kPa
Mobile phase :10%EtOAc/n-Hexane(w/w)
Flow rate : 5cm/min. (15.7ml/min)
Detector : UV254nm

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