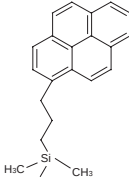
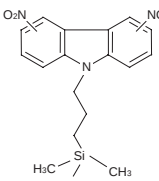
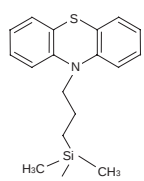
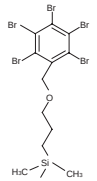


10. Fullerene Separation Columns

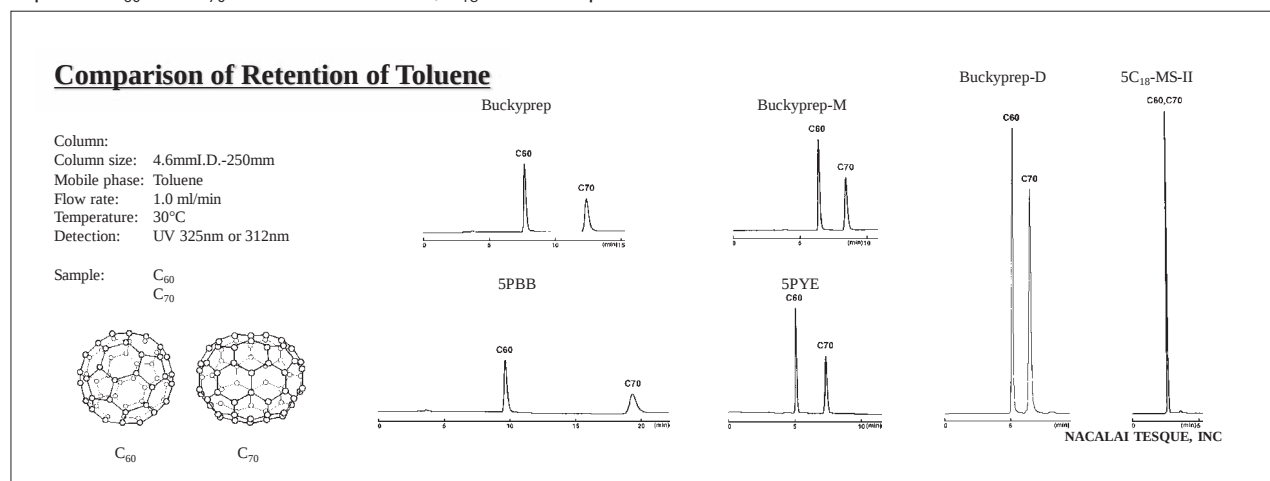
Separation of fullerenes, especially preparative scale separation, on conventional HPLC columns are always problematic due to the low solubility and low recovery rate of fullerenes. COSMOSIL offers a variety of columns designed for preparative scale separation of fullerenes including higher fullerenes, metallofullerenes and fullerene derivatives.

Specifications

Packing Material	Buckyprep	Buckyprep-D	Buckyprep-M	PBB
Silica Gel	High purity porous spherical silica			
Average Particle Size	5 μm			
Average Pore Size	120 $\text{\AA}$			
Specific Surface Area	300 m^2/g			
Bonded Phase Structure				
Bonded Phase	Pyrenylpropyl group	Nitro-carbazoyl group	Phenothiazinyl group	Pentabromobenzyl group
Bonding Type	Monomeric			
End-Capping Treatment	Near-perfect treatment		None	Near-perfect treatment
Carbon Content	17%	—	13%	8%
Features	• Standard column for fullerene separation	• Separation of fullerene derivatives	• Designed to separate metallofullerenes	• Designed for preparative separation of C_{60} , C_{70} .

Comparison of Retention

The figure below shows the retention time of C_{60} and C_{70} in toluene. Buckyprep, Buckyprep-M and PBB, and PYE nicely separate C_{60} and C_{70} . On the other hand, C_{18} cannot separate them in toluene.



Suggested Solvents for Fullerene Separation

Solvent	Features	Solubility of C_{60} (mg/mL)	Solvent	Features	Solubility of C_{60} (mg/mL)
Toluene	The most commonly used solvent	3.2	Acetonitrile	Weaker eluent than toluene. Recommended as a washing solvent Buckyprep-D	0.018
<i>n</i> -Hexane	Weaker eluent than toluene. Recommended for weakly retained fullerenes.	0.046	Chlorobenzene	Stronger eluent than toluene. Recommended for higher fullerenes	7.0
<i>n</i> -Heptane		—	<i>o</i> -Dichlorobenzene	Stronger eluent than chlorobenzene	27
Methanol		0.001	1,2,4-Trichlorobenzene	The strongest elution effect. Recommended as a washing solvent.	21.3
2-Propanol		—			

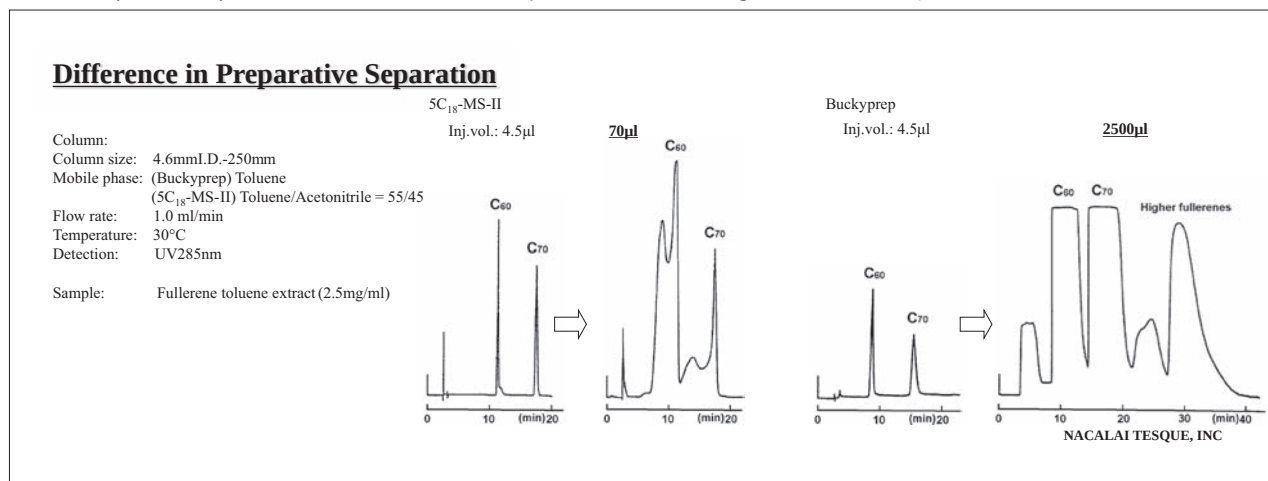
COSMOSIL Buckyprep



- Standard column for fullerene separation
- Excellent separation for higher and derivatized fullerenes

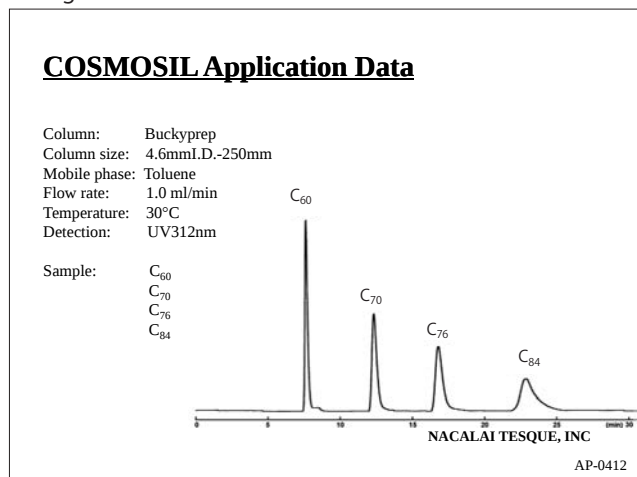
Difference in Preparative Separation

COSMOSIL Buckyprep can be used with toluene, the most commonly-used solvent in fullerene separation. Because tailing does not occur, you can inject about 35 times more sample, 2,500 μL (6.25 mg), than with a C_{18} column.

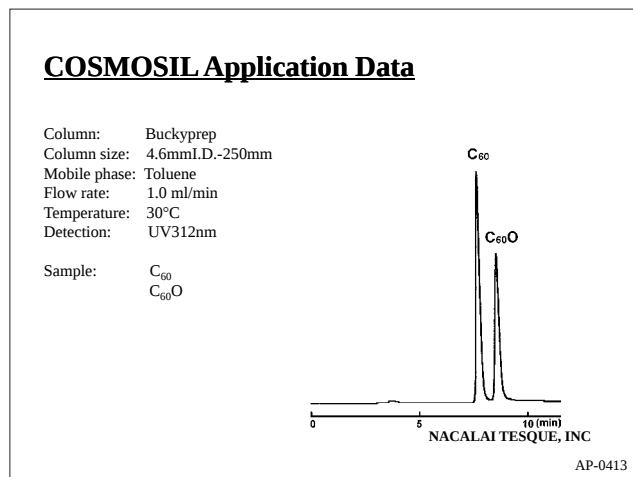


Applications

- Higher Fullerenes



- Oxidized Fullerenes



Ordering Information

- COSMOSIL Buckyprep Analytical / Preparative Columns (Particle Size: 5 μm)

Packed Column

I.D. x Length (mm)	Product Number
4.6 × 250	37977-61
10 × 250	37981-91
20 × 250	37982-81
28 × 250	34346-11

Guard Column

I.D. x Length (mm)	Product Number
4.6 × 10	37983-71
10 × 20	37984-61
20 × 50	34374-41
28 × 50	05871-21

COSMOSIL Buckyprep-D



- For preparative separation of derivatized fullerenes
- For separation of derivatized fullerenes such as C₆₀ indene used for organic thin-film solar cell

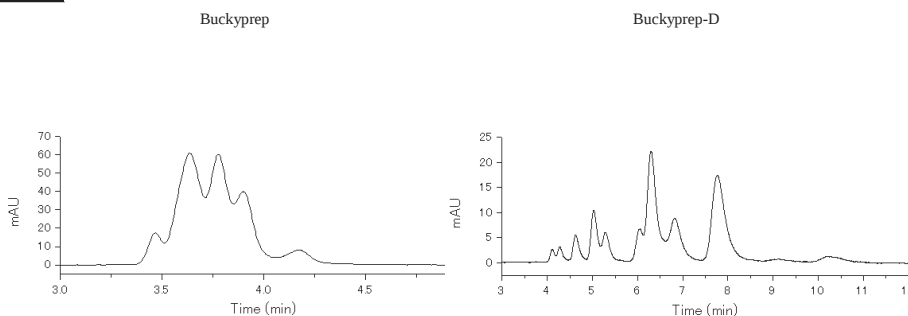
Applications

Buckyprep-D offers improved separation for C₆₀ indene, a derivatized fullerene, that has received much attention as an n-type semiconductor material for organic thin-film solar cells.

COSMOSIL Application Data

Column:
Column size: 4.6mm I.D. × 250mm
Mobile phase: Toluene
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV 325nm

Sample: C₆₀ [Indene]₂ (1.0mg/ml)
Inj. Vol.: 1.0μl



Data courtesy of Yusuke Tajima, Dr. Sci.
Organic Optoelectronics Laboratory, RIKEN (Institute of Physics and Chemistry)

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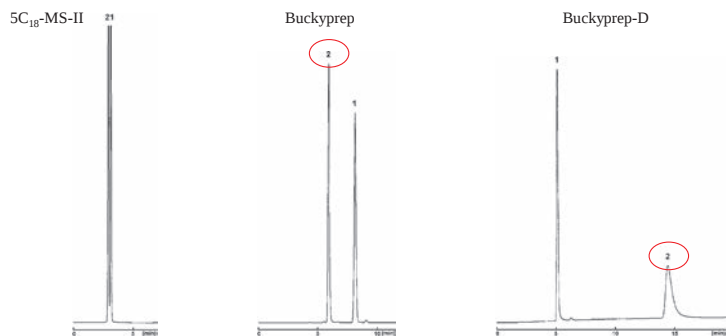
AP-1234

Buckyprep-D retains derivatized fullerenes longer than C₆₀. Therefore it is more suitable for preparative separation of derivatized fullerenes than our conventional Buckyprep column.

COSMOSIL Application Data

Column:
Column size: 4.6mm I.D. × 250mm
Mobile phase: Toluene
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV 325nm

Sample: 1; C₆₀ (0.25mg/ml)
2; [6,6]-Phenyl-C₆₁ Butyric Acid Methyl Ester [PCBM] (0.25mg/ml)
Inj. Vol. 1.0μl



NACALAI TESQUE, INC

AP-1232

Note

The baseline of COSMOSIL Buckyprep-D is less stable relative to other fullerene columns. To stabilize baseline, let acetonitrile run through for 10 minutes before analysis.

Ordering Information

- COSMOSIL Buckyprep-D Analytical / Preparative Columns (Particle Size: 5 μm)

Packed Column

I.D. x Length (mm)	Product Number
4.6 × 50	09646-61
4.6 × 250	09647-51
10 × 50	09648-41
10 × 250	09650-91
20 × 250	09651-81

Guard Column

I.D. x Length (mm)	Product Number
4.6 × 10	09611-01
10 × 20	09613-81
20 × 50	09614-71

COSMOSIL Buckyprep-M



- Different selectivity from Buckyprep
- Excellent separation for metallofullerenes

Applications

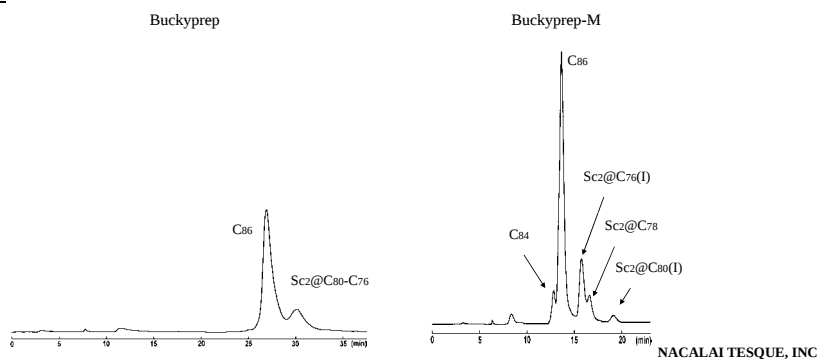
● Metallofullerenes

COSMOSIL Buckyprep-M is a phenothiazinyl-bonded silica-based column specifically designed for metallofullerene separation. Metallofullerenes are retained more strongly than other fullerenes on this column. COSMOSIL Buckyprep-M is also effective for the separation of higher fullerenes and fullerene derivatives.

COSMOSIL Application Data

Column:
Column size: 4.6mm I.D.-250mm
Mobile phase: Toluene
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV312nm

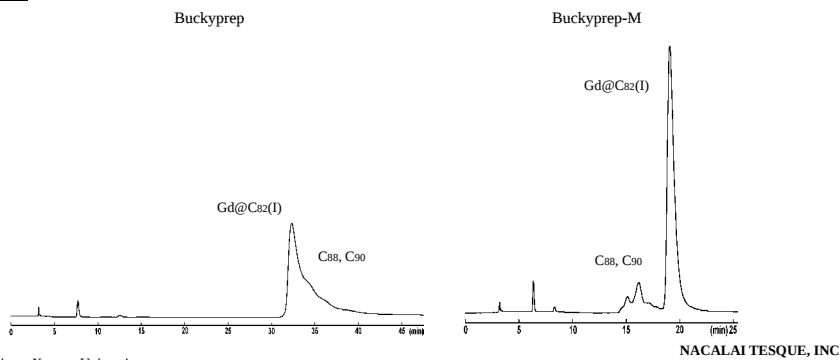
Sample: Sc₂@C₇₆(I)
Sc₂@C₇₈
Sc₂@C₈₀(I)
C₈₆



COSMOSIL Application Data

Column:
Column size: 4.6mm I.D.-250mm
Mobile phase: Toluene
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV312nm

Sample: Gd@C₈₂(I)



Ordering Information

● COSMOSIL Buckyprep-M Analytical / Preparative Columns (Particle Size: 5 μm)

Packed Column

I.D. x Length (mm)	Product Number
4.6 × 250	04138-71
10 × 250	04141-11
20 × 250	04142-01
28 × 250	05873-01

Guard Column

I.D. x Length (mm)	Product Number
4.6 × 10	04139-61
10 × 20	04140-21
20 × 50	34474-31
28 × 50	05872-11

COSMOSIL PBB



- Can be used with *o*-dichlorobenzene or carbon disulfide
- Suitable for preparative scale separation

Applications

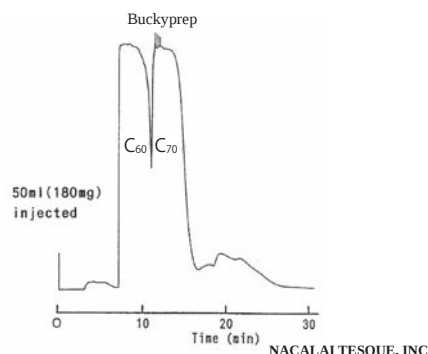
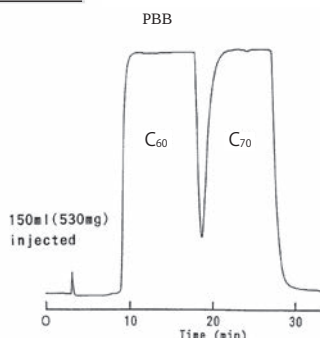
● Preparative Separation of Fullerene

The loading capacity of COSMOSIL PBB for C₆₀ and C₇₀ can be three times greater than COSMOSIL Buckyprep.

Comparison of Preparative Separation

Column: 20mm I.D.-250mm
Mobile phase: Toluene
Flow rate: 18 ml/min
Temperature: Room temperature
Detection: UV285nm

Sample: Crude fullerenes (3.5mg/ml)



Ordering Information

● COSMOSIL 5PBB Analytical / Preparative Columns (Particle Size: 5 μm)

Packed Column

I.D. x Length (mm)	Product Number
4.6×250	37980-01
10×250	37985-51
20×250	37986-41

Guard Column

I.D. x Length (mm)	Product Number
4.6×10	37987-31
10×20	37988-21
20×50	34375-31

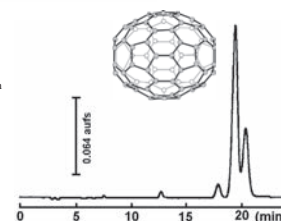
Fullerene Chromatogram Index

Fullerene Chromatogram Index includes more than 100 chromatograms. If you are interested in this index, please feel free to contact us. The online version is available at the website of The Fullerenes, Nanotubes and Graphene Research Society below.

Website:
http://fntg.jp/en/chromato_index_3.pdf

Fullerene Chromatogram Index

Sample: C78
CAS No.: 136316-32-0
Molecular formula: C78
Column: Buckyprep
Column size: 4.6 mm I.D.-250 mm
Mobile phase: Toluene
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV 285 nm
Attenuation: 0.64 au/fs
Sample conc.: 0.20 mg/ml
Injection volume: 10.0 μl



Data courtesy of
NACALAI TESQUE, INC