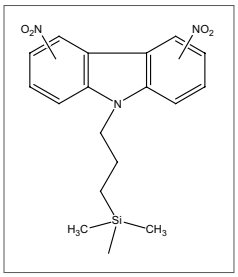
COSMOSIL

HPLC Column for Derivatized Fullerene Separation

COSMOSIL Buckyprep-D

- ***For preparative separation of derivatized fullerenes***
- ***For separation of derivatized fullerenes such as C_{60} indene used for organic thin-film solar cell***



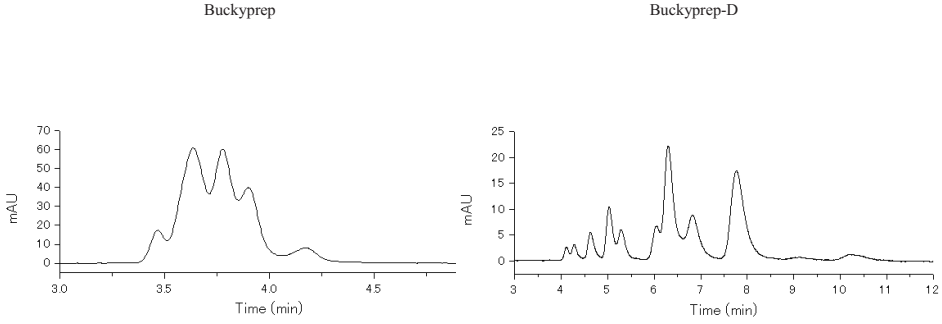
Application of C_{60} Indene

Buckyprep-D offers improved separation for C_{60} indene, a derivatized fullerenes, that has received much attention as the *n*-type semiconductor material of organic thin-film solar cell.

COSMOSIL Application Data

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

Sample: C_{60} [Indene]$_2$ (1.0mg/ml)
Inj.Vol.: 1.0µl



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

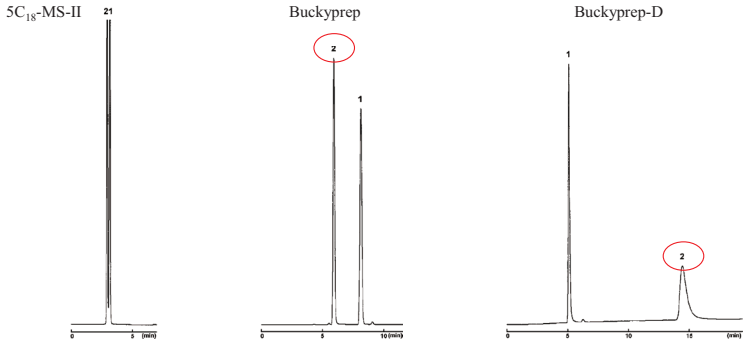
Comparison with Conventional Buckprep Columns

Buckyprep-D retains derivatized fullerenes longer than C_{60}. Therefore it is more suitable for preparative separation of derivatized fullerenes than conventional Buckyprep column.

COSMOSIL Application Data

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

Sample: 1; C_{60} (0.25mg/ml)
2; [*6,6*]-Phenyl-C_{61} Butyric Acid Methyl Ester [PCBM] (0.25mg/ml)
Inj.Vol. 1.0µl



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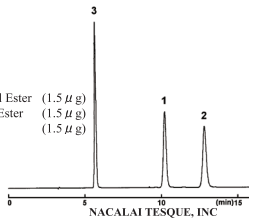
Buckyprep-D offers improved separation in toluene, a solvent with strong solubility for fullerenes (see Comparison with Conventional Type Columns in previous page.). It is necessary to mix in hexane with toluene when using the NPE column.
(See table below.).

COSMOSIL Application Data

Column: NPE
Column size: 4.6mmI.D.-250mm
Mobile phase: Toluene/ Hexane = 25/75
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV325nm

Sample:
1; [6,6]-Phenyl-C_{61} Butyric Acid Methyl Ester (1.5 μg)
2; [6,6]-Phenyl-C_{61} Butyric Acid Butyl Ester (1.5 μg)
3; C_{60} (1.5 μg)



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AP-1069

Table:
Solubility and boiling point of C_{60} in each solvent

Solvent	mg/ml	b.p. (°C)
Methanol	0.001	64.5
Acetonitrile	0.018	81.8
n-Hexane	0.046	68.7
Toluene	3.2	111
Chlorobenzene*	7.0	132
Carbon disulfide	12	46.3
1,2,4-Trichlorobenzene	21.3	213
o-Dichlorobenzene*	27	180

** R.S.Ruoff, et al., J. phy. Chem., 97, 3379 (1993)*

Application

• Fullerene di-adduct

COSMOSIL Application Data

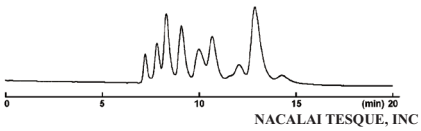
Column:
Column size: 4.6mmI.D.-250mm
Mobile phase: Toluene
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV 325nm

Sample: Fullerene di-adduct

Sample courtesy of Prof. Yutaka.Matsuo,
Department of Chemistry, The University of Tokyo

Buckyprep

Buckyprep-D



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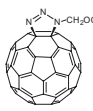
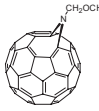
• Azafulleroid

COSMOSIL Application Data

Column:
Column size: 4.6mmI.D.-250mm
Mobile phase: Toluene
Flow rate: 1.0 ml/min
Temperature: 25°C
Detection: UV 310nm
Sample: MOM-triazolinofullerene
MOM-azafulleroid

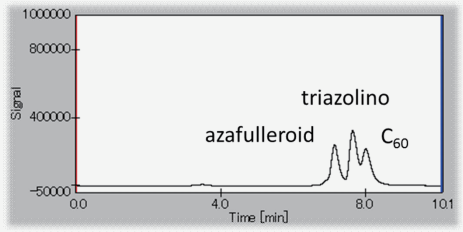


triazolino

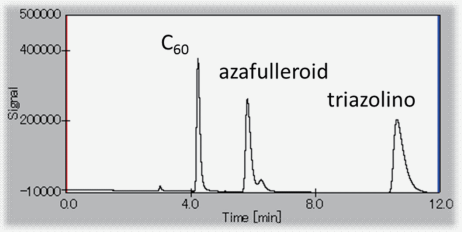


azafulleroid

Buckyprep



Buckyprep-D



Data courtesy of Assistant Prof. Naohiko Ikuma
Division of Applied Chemistry, Graduate School of Engineering, Osaka University

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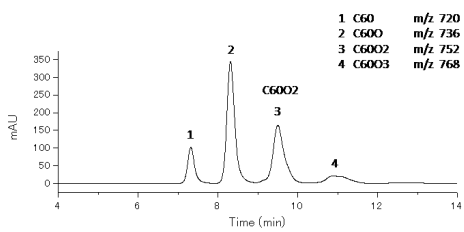
• Fullerene Oxid

COSMOSIL Application Data

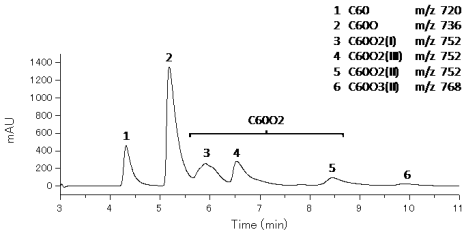
Column:
Column size: 4.6mmI.D.-250mm
Mobile phase: Toluene
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV 325nm

Sample: $C_{60}O_n$ (10mg/ml)
Inj.Vol.: 1.0μl

Buckyprep



Buckyprep-D



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

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Solution for Too Strong Retention on Buckyprep-D

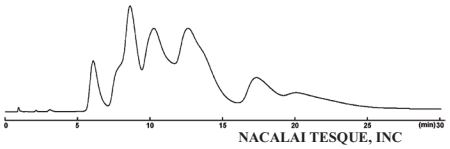
If the retention of derivatized fullerens on Buckyprep-D is too strong, shorten column length or add additives in toluene to decrease retention.

・Using 50 mm length column

COSMOSIL Application Data

Column: Buckyprep-D
Column size: 4.6mmI.D.-50mm
Mobile phase: Toluene
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV325nm

Sample: [6,6]-Diphenyl-C_{62} Bis (Butyric Acid Methyl Ester)
[Bis[60]PCBM] (0.50mg/ml)
Inj.Vol.: 10µl



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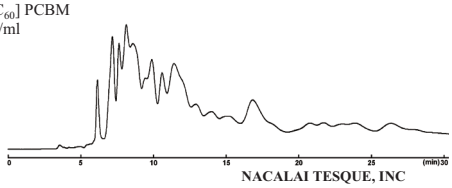
・Adding methanol

COSMOSIL Application Data

Column: Buckyprep-D
Column size: 4.6mmI.D.-250mm
Mobile phase: Methanol/ Toluene = 1/99
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV325nm

Sample: Tris [C_{60}] PCBM
Sample conc.: 1.0mg/ml
Inj.Vol.: 10.0µl



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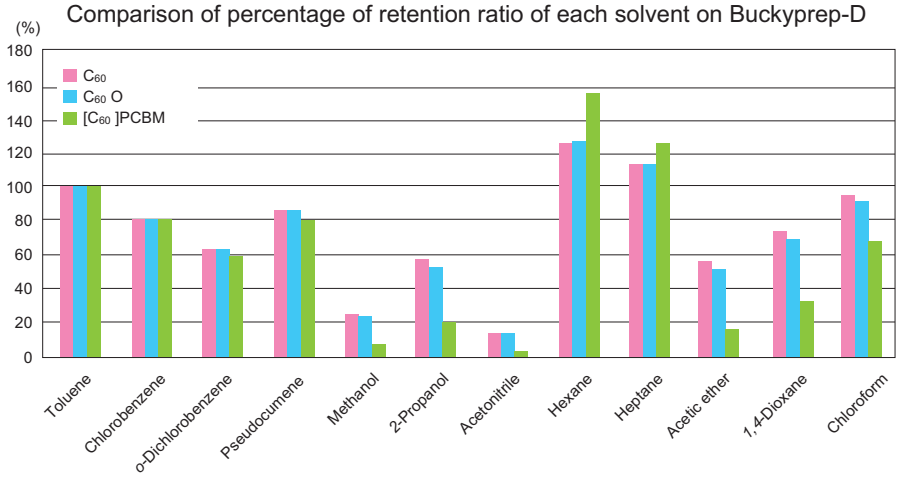
Adjustment of Retention by Mobile Phase

・Comparison of each solvent

Adding hexane or heptane to toluene increases retention on Buckyprep-D, while adding methanol or acetonitrile decreases retention. The effect of solvents on retention is the opposite to the conventional Buckyprep column.

Comparison of percentage of retention ratio of each solvent on Buckyprep-D



Y-axis: Percentage of capacity factor k' when it is 100% toluene
X-axis: 10% of each solvent added into toluene

・Adding methanol decreases retention

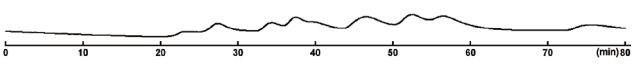
Retention may decrease when a polar solvent is added on Buckyprep-D. For example, by increasing the methanol concentration to 1%, the retention dramatically decreases.

COSMOSIL Application Data

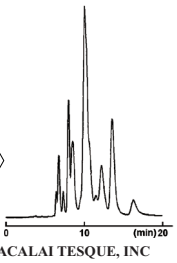
Column: Buckyprep-D
Column size: 4.6mmI.D.-250mm
Mobile phase:
Flow rate: 1.0 ml/min
Temperature: 30°C
Detection: UV 325nm

Sample: [6,6]-Diphenyl-C_{62} Bis(Butyric Acid Methyl Ester)
(0.5mg/ml)
Inj.Vol.: 5.0µl

Toluene



Methanol/ Toluene = 1/99



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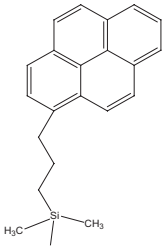
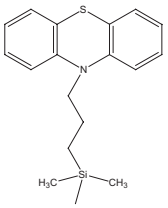
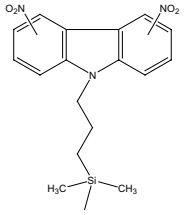
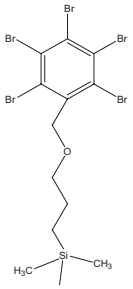
Buckyprep Series

Standard column for fullerenes → COSMOSIL Buckyprep
For separation of metallofullerenes → COSMOSIL Buckyprep-M
For separation of derivatized fullerenes→ COSMOSIL Buckyprep-D

Specification of COSMOSIL Fullerene Columns

Packing Material	Buckyprep	Buckyprep-M	Buckyprep-D	PBB
Silica Gel	High Purity Porous Spherical Silica			
Average Particle Size	5 µm			
Average Pore Size	approx. 120 Å			
Specific Surface Area	approx. 300 m^2/g			
Bonded Phase Stracture				
Bonded Phase	Pyrenylpropyl group	Phenothiazinyl group	Nitro-carbazoyl group	Pyrenylethyl group
End-capping Treatment	near-perfect	none	near-perfect	near-perfect
Features	Standard column for fullerenes separation	For separation of metallofullerenes	For separation of fullerene derivatives	For preparative separation of C_{60}, C_{70}

Features of Each Solvent for Fullerenes Separation

Solvent	Feature	Solubility of C_{60} (mg/ml)
Toluene	The most commonly used solvent	3.2
n-hexane		0.046
n-heptane	Weaker eluent than toluene	-
Methanol*		0.001
2-propanol*		-
Acetonitrile*	Weaker eluent than toluene. Recommended as a wash solvent for Buckyprep-D	0.018
Chlorobenzene	Stronger eluent than toluene	7.0
o-dichlorobenzene	Stronger eluent than chlorobenzene	27
1,2,4-trichlorobenzene	The strongest eluent. Recommended as a wash solvent	21.3

* These are stronger eluent on Buckyprep-D, opposite to Buckyprep.

Attenion: Use HPLC Grade solvent or solvents after filtration or distillation. Except for alkali aqueous and strong acidic solutions, other solvents can be used (water-free pyridine and others). Depending on solvents, pay attention to high pressure caused by high solvent viscosity.

Note: Baseline of Bukyprep-D is less stable relative to other fullerene columns. To stabilize baseline, let acetonitrile run through for 10 minutes before analysis.

Ordering Information

Product Name	Column Size	Product No.
COSMOSIL Buckyprep-D Packed Column	4.6 mm I.D. x 50 mm	09646-61
	4.6 mm I.D. x 250 mm	09647-51
	10.0 mm I.D. x 250 mm	09650-91
	20.0 mm I.D. x 250mm	09651-81

Product Name	Column Size	Product No.
COSMOSIL Buckyprep-D Guard Column	4.6 mm I.D. x 10 mm	09611-01
	10.0 mm I.D. x 20 mm	09613-81
	20.0 mm I.D. x 50 mm	09614-71

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The quality for certainty.

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