

For High Performance Liquid Chromatography

COSMOSIL[®] PACKED COLUMNS for fullerenes

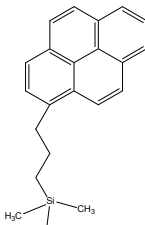
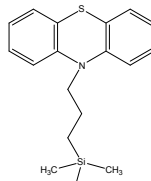
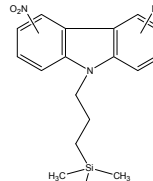
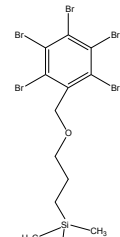
1. INTRODUCTION

Thank you for purchasing our COSMOSIL Packed Columns for fullerenes. Please read this manual carefully before use to ensure maximum efficiency and a long column life.

2. TYPES OF STATIONARY PHASES AND THEIR CHARACTERISTICS

COSMOSIL Packed Columns for fullerenes are packed with the silica gel that bonded specially designed stationary phase. Our columns are shown in Table 1. Please choose the column suitable for your samples.

Table 1. Types of stationary phases and their characteristics

Packing material	Buckyprep	Buckyprep-M	Buckyprep-D	PBB
Silica gel	High purity porous spherical silica			
Average particle size	5 μ m			
Average pore size	approx. 120Å			
specific surface area	approx. 300m ² /g			
Stationary phase				
	Pyrenylpropyl group	Phenothiazinyl group	Nitrocarbazolyl group	Pentabromobenzyl group
End capping treatment	Treatment	None	Treatment	Treatment
Feature	Standard column for fullerene separation.	Designed to separate metallofullerenes.	Designed to separate fullerenes derivatives.	Designed for preparative separation of C ₆₀ , C ₇₀ .

3. TROUBLESHOOTING

Table 2. Suggested solvents

Solvent	Feature	Solubility for C ₆₀ (mg/ml)
Toluene	It is generally used to separation of fullerenes.	3.2
<i>n</i> -Hexane	Weak eluent than toluene.	0.046
<i>n</i> -Heptane		**
Methanol*		0.001
2-Propanol*		**
Acetonitrile*	Weak eluent than toluene. It is used for washing solvent before analysis with Buckyprep-D.	0.018
Chlorobenzene	Stronger eluent than toluene.	7.0
<i>o</i> -Dichlorobenzene	Stronger eluent than chlorobenzene.	27
1,2,4-Trichlorobenzene	Strongest eluent. It can be used as a washing solvent for higher fullerenes.	21.3

* In Buckyprep-D, they are stronger eluent than toluene.

(Note) Use them after filtration or distillation, if they are not for HPLC

4. CARE AND USE

1. Buckyprep-D has the fault of being hard to stabilize the baseline compared with other columns. In order to stabilize the baseline, it is effective to wash acetonitrile about 10 minutes before analysis.
2. Avoid mechanical shocks to the column.
3. Connect the column according to the flow direction indicated on the label.
4. Keep pressure under 20MPa (15MPa in case of more than 10mmI.D). Take special care when using highly viscous mobile phase.
5. Elute the column with 20-30 ml mobile phase before connecting to the detector.
6. Use scrupulously degassed mobile phase. Air bubbles generate detection noise and accelerate column deterioration.
7. After analysis, wash the column with acid free, alkaline free and halogen free solvent. Store it tightly plugged.
8. Filter the sample before injection. Avoid precipitation at injection.
9. Removal of the end filters or change of the end-fittings will result in low performance of the column.
10. Do not tighten nuts more firmly than necessary.
11. Establish perfect chromatography conditions by experimenting with an analytical column of the same packing material before employing a preparative column. Pay attention to impurities with longer retention time than your sample.
12. In order to maximize column performance, minimize the dead volume of mobile phase in the equipment by shortening and/or narrowing the width of tubing.
13. Maintain constantly column and tubing temperature.
14. Avoid injecting air, changing flow rate rapidly and changing mobile phase at high flow rate.
15. Insoluble matters from the pumping system, mobile phase, or samples trapped in the filter at the inlet of the column may increase the pressure.
16. We recommend using guard column to protect from irreversible adsorption on the packing material, clogging of the end filter by insoluble matters, or rapid pressure increase.
17. We recommend keeping the chromatography conditions constant because frequent changes of mobile phases shorten column life.

5. TROUBLESHOOTING

Problem	Possible Cause	Solution
Increase of pressure	Clogging of the end filter Clogging of the packing material Precipitation in the column	(1) (2) (2)
Poor resolution	Contamination of packing material Disorder of packing material	(2)(3) Unregenerable
Split peak	Void in the column	Unregenerable
Unstable baseline	Contamination of packing material Contamination of mobile phase	(2)(3) (4)

- (1) Disconnect column from the detector. Wash with mobile phase through the column in reverse direction at half flow rate for 30 minutes.
- (2) Wash the column with high solubility solvent.
- (3) Wash the column with stronger eluent (ex. 1,2,4-trichlorobenzene).
- (4) Use the new solvent.

4. WARRANTY

Nacalai Tesque will replace defective columns reported within 2 weeks of receipt. Nacalai Tesque approves return in case of:

- (1) Damage during transportation caused by our incomplete packing.
- (2) Theoretical plate number measured according to the test method specified in the Inspection Report is significantly lower than guaranteed.
(Please note that the plate number decreases when using apparatus with large dead volume or injecting big amount of sample.)

We cannot accept claims for deterioration of column performance caused by taking off the end filters or end-fittings or long shelf life. Return shipment is unacceptable unless we have given prior permission and shipping instructions.