No.A-008E

Recycling Preparative HPLC
LaboACE LC-5060

Related Product : Recycling Preparative HPLC Series

Separation of compounds that are inseparable by Silica columns 3

Keyword:
GPC Column, SEC Column, Size Exclusion Chromatography, Recycling Preparative HPLC

Introduction

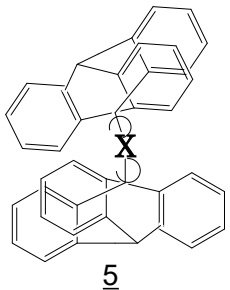
For substances that are unstable against silica-based fillers or that were inseparable by TLC, the combination of GPC (SEC) column and Recycling Preparative HPLC is often a very effective solution.

Here is an example of such studies by a professional who uses our Recycling Preparative HPLC systems.

Experiment and Results

In synthesizing bis(triptycyl) sulfide, disulfide form is also obtained as a byproduct (5).

It is not possible to separate them by silica gel columns which utilize the difference in polarity because the -C-S-C- bond and –C-S-S-C- bond are deep inside the molecules. So we tried to separate them using GPC column.



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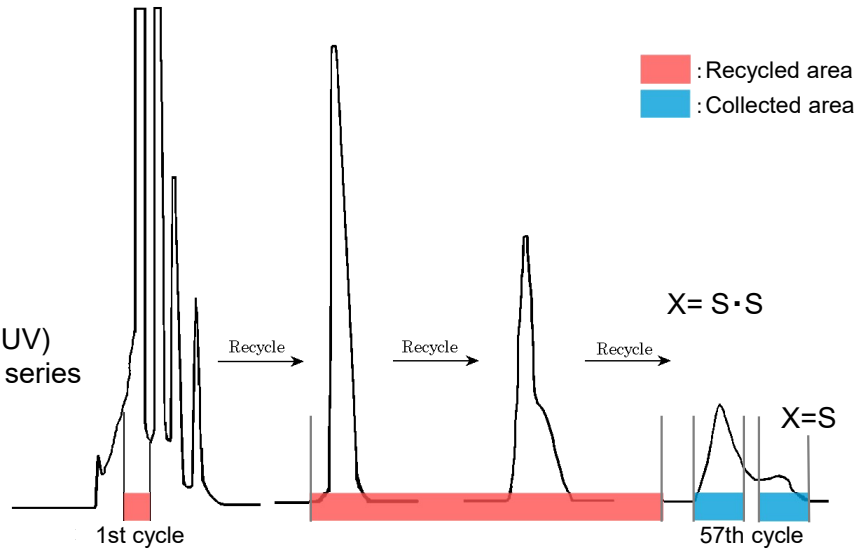
Instrument	:	LC-908 (Detector : UV)
Column	:	JAIGEL-1H + 1H in series
Solvent	:	Chloroform
Flow rate	:	3.0 mL/min



Separation of bis(triptycyl) compounds

Conclusion

The two compounds were completely separated at the 57th cycle using two JAIGEL-1H columns. It usually takes 10 cycles or so to separate compounds with this kind of molecular weight and with a difference of one sulfur atom. It is presumed that it took as many cycles as 57 this time because the length of one additional “-S-” bond is not so clearly recognized in GPC column.

References

Yuzo Kawada, Joji Ishikawa, Hiroshi Yamazaki, Gen Koga, Shigeru Murata, and Hiizu Iwamura, Tetrahedron Letters, Vol. 28, No.4, pp 445-448 (1987)

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