The Application of Column Stacking in the Purification of Organic Optoelectronic Materials

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Chromatography Application Note AN021

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Introduction

Column stacking is a commonly used method to improve resolution in flash chromatography. In our previously published application note The Improvement of the Resolution by Column Stacking and Its Application in the Compound Purification, we have elaborated in principle how to improve resolution by column stacking and its application in the purification of a synthetic sample. In this post, an organic optoelectronic material was utilized as the sample to demonstrate the advantage of column stacking to achieve higher resolution.

Experimental Section

The sample used in the experiment was a synthetic crude product of organic optoelectronic materials which was provided by a company focusing on OLED new material research and development. Regarding the application of SepaBean™ machine combined with SepaFlash™ series flash cartridges for the rapid purification of organic optoelectronic materials, please refer to another previously published application note The Application of SepaBean Machine in the Field of Organic Optoelectronic Materials.

The sample was first analyzed by thin layer chromatography (TLC). The sample was spotted on a TLC plate and then placed in a developing tank filled with n-hexane/ethyl acetate as the developing solvent. As shown in Figure 1, the sample spot was very close to other impurity spots, indicating low sample purity and relatively difficult purification of the target product

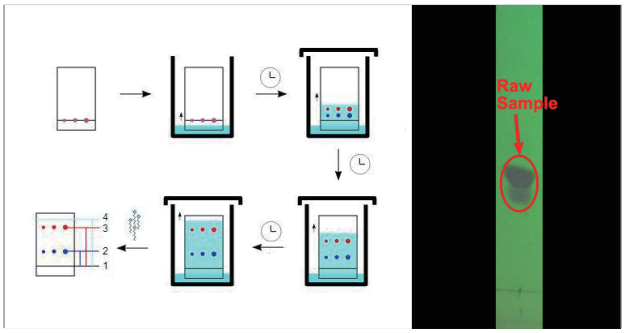

Figure 1. The schematic diagram of TLC principle (left part) and the TLC development result of the sample (right part).

The SepaFlash™ normal phase flash cartridges were utilized by the researchers at Application R&D Center of Santai Technologies for the purification of the sample. To improve the resolution for the sample, column stacking was adopted by stacking two SepaFlash™ Standard Series flash cartridges with another solid loading cartridge on the top. 100 mg of the sample was dissolved in DCM and then mixed with 1 g of silica gel. Afterwards the sample mixture was dried by rotary evaporation and loaded on the solid loading cartridge. The experimental setup of the flash purification is listed in Table 1.

Instrument	SepaBean™ machine 2	
Cartridges	12 g SepaFlash™ Standard Series flash cartridge (irregular silica, 40 - 63 μm, 60 Å, Order number: S-8101-0012) 12 g SepaFlash™ HP Series flash cartridge (High-capacity spherical silica, 25 μm, 50 Å, Order number: SW-2102-012-SP(H))	
Wavelength	220 nm (detection); 254 nm (monitoring)	
Mobile phase	Solvent A: N-hexane Solvent B: Ethyl acetate	
Flow rate	15 mL/min	
Sample loading	100 mg of crude sample	
Gradient	Time (min)	Solvent B (%)
	0	0
	30	5
	43	5
	55	9
	56	100
	65	100

Table 1. The experimental setup for flash purification.

Results and Discussion

The flash chromatogram of the sample separated by two stacking Standard Series flash cartridges was shown in Figure 2.

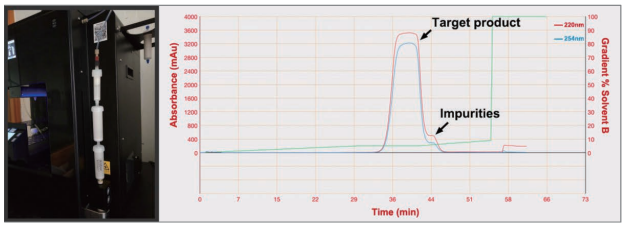

Figure 2. The flash chromatogram of the sample on two stacking Standard Series flash cartridges.

As shown in Figure 2, the target product and impurities failed to reach baseline separation, indicating sufficient resolution cannot be obtained by stacking two Standard Series flash cartridges. To improve the resolution, the researchers then tried to use SepaFlash™ HP Series flash cartridges which are pre-packed with smaller particle sized silica. Two HP Series flash cartridges were stacked in series for the sample purification while other experimental conditions were kept the same. The flash chromatogram was shown in Figure 3.

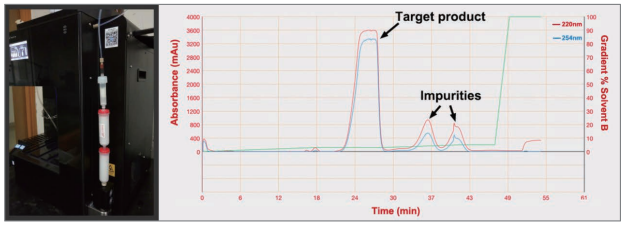

Figure 2. The flash chromatogram of the sample on two stacking HP Series flash cartridges.

As shown in Figure 3, the target product and impurities were separated at baseline level with higher resolution, indicating the resolution meeting the requirement could be achieved by stacking HP Series flash cartridges in series. To further validate the purification results, the collected fractions corresponding to the target product and the impurities were sampled for TLC identification. As shown in Figure 4, the target product and the impurities were well separated and could be used in the following research and development.

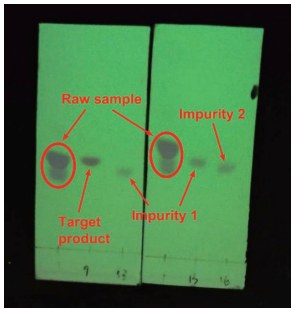

Figure 4. The TLC identification results of the samples corresponding to raw sample and purified fractions.

About the SepaFlash™ HP Series flash cartridges

There are a series of the SepaFlash™ HP Series flash cartridges with different specifications from Santai Technology (as shown in Table 2).

Item Number	Column Size	Flow Rate (mL/min)	Max.Pressure (psi/bar)
SW-2102-004-SP(H)	4 g	15-30	400/27.5
SW-2102-012-SP(H)	12 g	25-50	400/27.5
SW-2102-025-SP(H)	25 g	25-50	400/27.5
SW-2102-040-SP(H)	40 g	30-60	400/27.5
SW-2102-080-SP(H)	80 g	40-80	350/24.0
SW-2102-120-SP(H)	120 g	45-90	300/20.7
SW-2102-220-SP(H)	220 g	60-120	300/20.7
SW-2102-330-SP(H)	330 g	60-120	250/17.2

Table 2. SepaFlash™ HP Series flash cartridges. Packing materials: High-capacity spherical silica, 25 μm, 50 Å.

For further information on detailed specifications of SepaBean™ machine, or the ordering information on SepaFlash™ series flash cartridges, please visit our website: http://www.santaitech.com/en/index.php .

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