05 SepaFlash® Large Purification Products for Hundreds of Grams of Samples

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Abstract

With the continuous development of medium/low pressure preparative chromatography instruments, more and more researchers tend to use automatic instruments to separate and purify samples owing to its great convenience and efficiency. However, due to the limitation of instrument configuration or other factors, researchers usually handle with small amounts of sample (less than 10 g) by the instrument. When handling with dozens or even hundreds of grams of samples, column packing, eluting and collecting by manual methods are always employed by researchers (as shown in Figure 1).

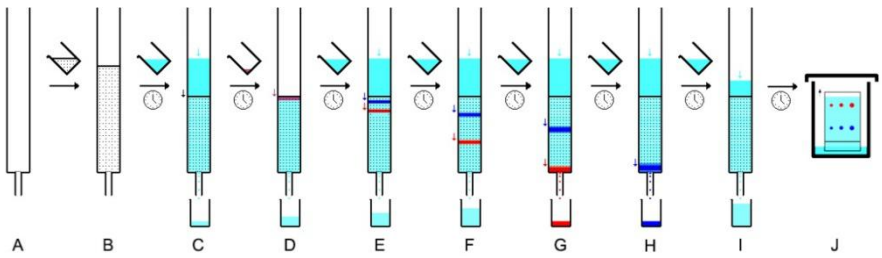

Figure 1. Schematic diagram of traditional manual column chromatography. A–B: Column packing. C: Column equilibrium. D. Sample loading. E-F: Elution. G-H: Fraction collection. I: Column cleaning. J: Detection by TLC.

To improve the working efficiency of the researchers, SepaFlash® Large purification cartridge series are developed. The maximum packing size is up to 3 kg of silica gel, making the large cartridge able to purify hundreds of grams of crude samples in single run. In this application note, an 800g-sized SepaFlash® large cartridge pre-packed with ultra-pure silica gel (Product number: S-5101-0800) combined with a preparative flash chromatography system was utilized for the purification of 100 g sample. The results showed good peak resolution as well as higher sample loading capacity comparing with manual method, suggesting an efficient solution for large amount sample purification.

Experimental

➢ Cartridge installation and equilibrium

An 800g SepaFlash® Standard Series flash cartridge (Order number: S-5101-0800) was used in the separation experiment. The inlet and outlet for the mobile phase were properly connected with tubes according to the ***Guidelines on Santai Adaptor Kit for 800g and 1600g Flash Columns***. Afterwards n-hexane was used to equilibrate the cartridge until the silica gels packed in the cartridge were completely wetted.

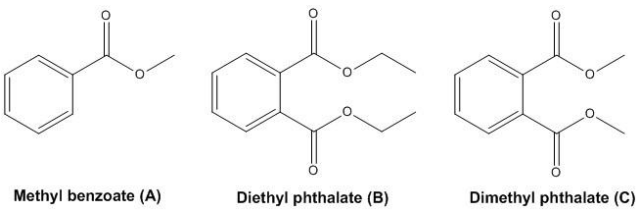

Figure 2. The chemical structure of Sample A, B and C.

Solid sample loading

A 100 g of sample mixture (as shown in Figure 2) was dissolved in ethyl acetate and absorbed onto 150 g of silica gel of 100-200 meshes. Ethyl acetate was removed by vacuum and the absorbed sample placed in a 220g iLOK™ empty cartridge for sample loading (as shown in Figure 4). Sample was then eluted according to the parameters as shown in Table 1.

Table 1. The experimental parameters.

Instrument	A preparative flash chromatography system	
Cartridges	800g SepaFlash® Standard Series flash cartridge (UltraPure irregular silica, 40-63μm, 60Å, Order number: S-5101-0800)	
Wavelength	254 nm (detection), 280 nm (monitoring)	
Sample	100 g mixture of Sample A (5.0 g), B (6.0 g) and C (89.0 g)	
Mobile phase	Solvent A: N-hexaneSolvent Solvent B: Ethyl acetate	
Flow rate	120 ml/min	
Gradient	Solvent B (%)	Time (min)
	0	0
	0	3
	7	4
	10	13
	12	14
	25	52
	25	56

Results and Discussion

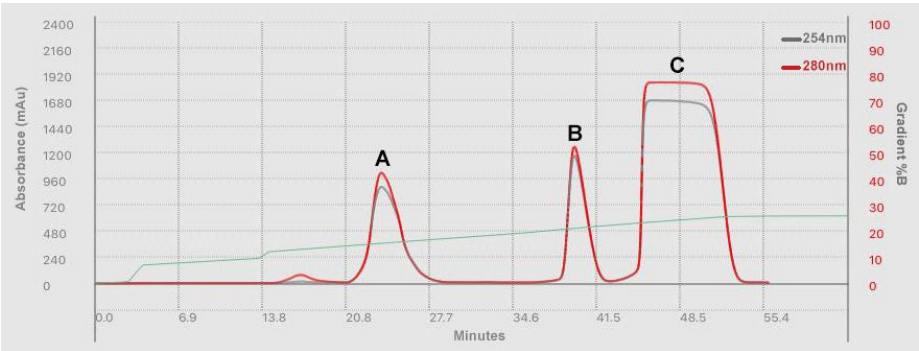

Figure 3. The chromatogram of the sample mixture in an 800g SepaFlash® Standard Series flash cartridge.

As shown in Figure 3, the large amount of the sample mixture (about 100 g) was successfully separated and purified in a single run.

Comparing with manual packed columns, the SepaFlash® cartridges pre-packed by automated instruments have better peak resolution as well as larger sample loading capacity.

Results and Discussion

In traditional manual separation by glass column, much time was spent during the procedure from glass column packing to manual fraction collection then frequently fraction confirmation by thin layer chromatography (TLC).

In contrast, much more human resources could be saved with the help of commercial flash cartridge combined with automated flash chromatography system.

Furthermore, the programmable gradient elution not only reduces the solvent consumption but also greatly decreases the solvent amount in the collected fractions, which in result reduces post treatment time.

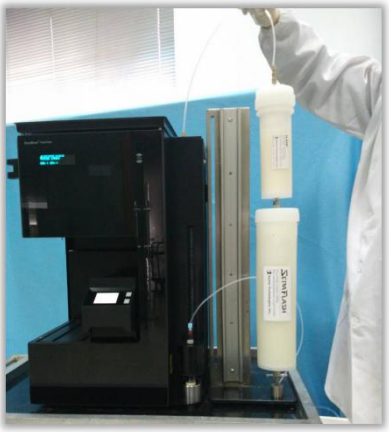

Figure 4. The experimental setup.

Recommended Flash Cartridges

Table 2. SepaFlash® Standard Series cartridges

Item Number	Column Size	Flow Rate (mL/min)	Max.Pressure (psi/bar)
S-5101-0004	4 g	15-40	300/20.7
S-5101-0012	12 g	30-60	300/20.7
S-5101-0025	25 g	30-60	300/20.7
S-5101-0040	40 g	40-70	300/20.7
S-5101-0080	80 g	50-100	200/13.8
S-5101-0120	120 g	60-150	200/13.8
S-5101-0220	220 g	80-220	150/10.3
S-5101-0330	330 g	80-220	150/10.3
S-5101-0800	800 g	100-300	100/6.9
S-5101-1600	1600 g	200-500	100/6.9
S-5101-3000	3000 g	200-500	100/6.9

Figure 5. SepaFlash® Standard Series cartridges.

Figure 6. SepaFlash® Standard Series 3kg cartridges.

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