

Higher Sample Loading Capacity, Better Performance – The Application of SepaFlash™ Ruby Series High Resolution Cartridges



Chromatography Application Note
AN022

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Introduction

In the daily separation and purification work of organic synthesis researchers, for samples with less difficulty in separation, normal phase separation with cartridges packed with irregular silica gel of 40 – 63 μm in particle size is usually used to meet the requirements of purification. However, for samples that are difficult to separate, or where impurities in the sample need to be collected for further analysis, higher requirements are imposed on the normal phase flash column packed with silica gels. Silica gels with smaller particle size and larger surface area have obvious advantages. Their better resolution and higher sample loading capacity make them a satisfactory solution for customers. In this post, a synthetic drug intermediate was used as the sample. Parallel comparison experiments were performed using three kinds of flash cartridges: SepaFlash™ Ruby High Resolution cartridge, SepaFlash™ Standard Series cartridge as well as a well-known brand flash cartridge. The results suggested SepaFlash™ Ruby High Resolution cartridge has better performance than other two cartridges, providing a powerful guarantee for the purification of complex samples.

Experimental Section

Experimental Section

The sample used in this post was an intermediate from a synthetic reaction. The synthetic scheme of the final step was shown in Figure 1.

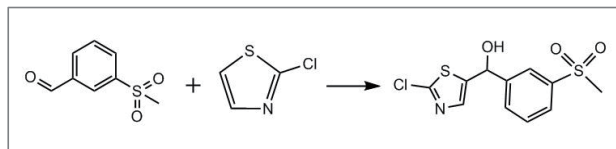


Figure 1. The final step in the synthetic scheme of the drug intermediate which was used as the sample.

TLC identification results of the sample

The sample was firstly identified by thin layer chromatography (TLC). The development solvent system was petroleum ester (PE) and ethyl acetate (EA) ($v/v = 1:1$). According to the TLC identification result which was shown in Figure 2, the R_f value of the target product spot was 0.4. Two impurity spots with the R_f value of 0.5 and 0.6 respectively were very close to the target product spot. Regarding the case of the target product spot was very close to other impurity spots ($\Delta R_f < 0.1$), higher demand was imposed for the chromatography material used in subsequent purification procedure.

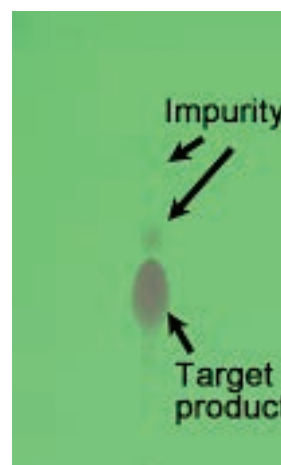


Figure 2. TLC identification result of the sample.

Flash purification of the sample

In this post, three flash cartridges of different specifications were used. The specifications of these cartridges were shown in Table 1.

Cartridge	A well-known brand product	SepaFlash™ Standard Series	SepaFlash™ Ruby Series
Silica type	Spherical, 20 - 40 μm	Irregular, 40 - 63 μm	Spherical, 25 μm
Pore size	60 Å	60 Å	50 Å
Surface area	N/A	500 m ² /g	700 m ² /g
Cartridge size	12 g	12 g	12 g
Suggested sample loading amount	60 mg - 1.2 g	12 mg - 1.2 g	12 mg - 3.6 g
Suggested flow rate	16.8 - 30 mL/min	30 - 60 mL/min	25 - 50 mL/min

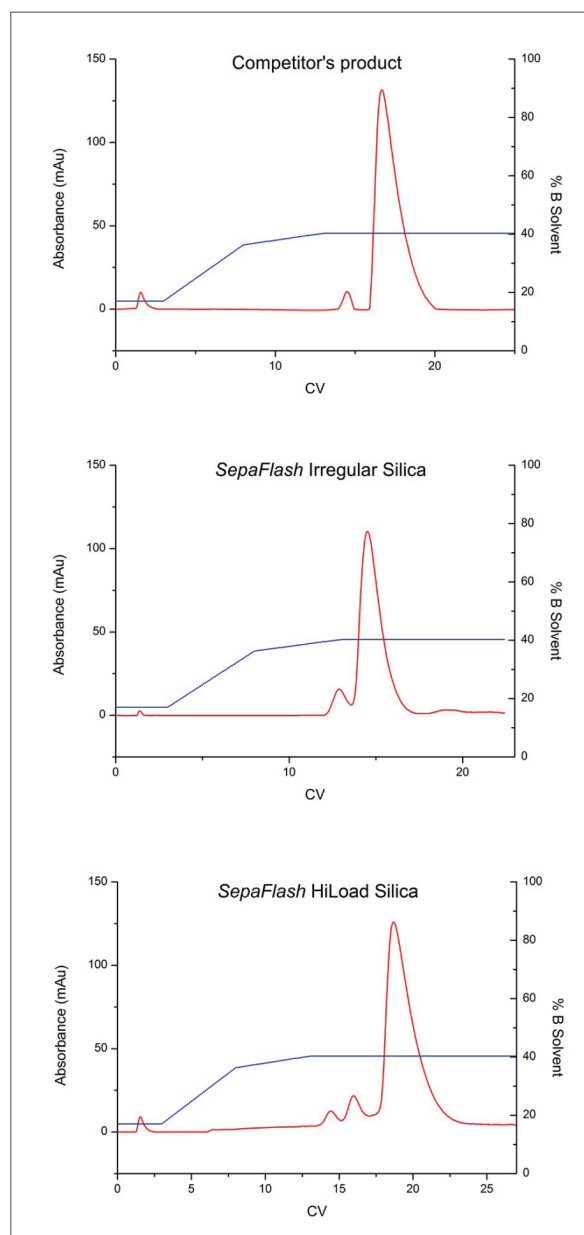
Table 1. Specifications of flash cartridges used in this study.

The R_f value of the target product and main impurities could be calculated according to the TLC identification result. Then input the TLC information on the related page of SepaBean™ App installed on iPad controlling SepaBean™ machine. Optimized elution gradient will be automatically generated by SepaBean™ App. The experimental setup of flash chromatography for the sample was shown in Table 2.

Instrument	SepaBean™ machine 2	
Cartridges	12 g well-known brand flash cartridge 12 g SepaFlash™ Standard Series cartridge (Order number: SW-5101-012) 12 g SepaFlash™ Ruby Series cartridge (Order number: SW-2102-012-SP(H))	
Wavelength	280 nm	
Mobile phase	Solvent A: N-hexane Solvent B: Ethyl acetate	
Flow rate	30 mL/min	
Sample loading	250 mg	
Gradient	Time (CV)	Solvent B (%)
	0	5
	3	5
	8	40
	12	45
	25	45

Table 2. The experimental setup for flash purification.

Results and Discussion



The flash chromatograms of the sample on different flash cartridges were shown in Figure 3. As shown in Figure 3, higher resolution for the impurities in the sample was achieved by SepaFlash™ Ruby Series cartridge. The two impurities with very close R_f value were separated at baseline level. The results indicated that SepaFlash™ Ruby Series cartridges have better performance than other silica cartridges. Thus they are ideal for fine purification of complex samples which might be difficult to purify by regular silica cartridges. Furthermore, under the premise of meeting the requirements of product purity and yield, higher sample loading amount could be achieved on SepaFlash™ Ruby Series cartridges, thereby improve the work efficiency as well as save a large amount of solvents and time.

About the SepaFlash™ Ruby Series flash cartridges

There are a series of the SepaFlash™ Ruby 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™ Ruby 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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