SANTAI TECHNOLOGIES

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The Purification of Alkaline Compounds by SepaFlash® Alumina Cartridges

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Abstract

In modern flash preparative chromatography the separation result is mainly determined by the choice of the column packing material. However, there are a wide range of column packing material could be selected. Therefore the most suitable packing material should be carefully considered with respect to the samples of different characteristics. The silica- or alumina-based packing materials are commonly used in flash preparative chromatography. For alkaline samples, flash cartridge pre-packed with silica usually shows poor separation results.

In the application note, a SepaFlash® E Series silica cartridge as well as an alumina cartridge was utilized for the separation and purification of alkaline compounds under the same chromatographic conditions. The results showed that the two alkaline compounds were failed to be separated due to the irreversible adsorption to the silica stationary phase. As a contrast, the sample mixture was successfully separated and purified in the alumina cartridge with suitable retention time as well as good resolution, suggesting an effective solution for the purification of these alkaline compounds.

Experiments- Sample

The sample was a mixture of 2-aminopyridine and 4-aminoisoquinoline (as shown in Figure 1). Both of them have relatively strong alkalinity.

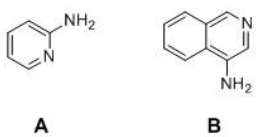

Figure 1. The chemical structure of two alkaline compounds.

Instruments and Consumables

The experiment was performed on a flash preparative liquid chromatography system (PF430, Interchim). The flash cartridges used in the experiment were a 40g SepaFlash® E Series silica cartridge as well as a 40g SepaFlash® E Series neutral alumina cartridge, both from Santai Technologies, Inc.

Experimental

a.Sample preparation

110 mg of 2-aminopyridine or 4-aminoisoquinoline was dissolved in 20 ml ethyl acetate, respectively. 2 g of neutral alumina-based packing material were added into the solution for adsorbing the sample onto alumina (for silica cartridge, silica-based packing material was used as the adsorption material). Then the solution was concentrated on a rotary evaporator.

b.Sample purification

The 40g neutral alumina cartridge or silica cartridges was equilibrated by n-hexane for 5 min. Then the sample prepared previously was loaded onto the flash cartridge and further eluted by the mobile phase of n-hexane/ethyl acetate with different gradient profiles. The flow rate was set to 30 ml/min. The experimental parameters were summarized in Table 1.

Table 1. The experimental parameters.

Instrument	Interchim PF430	
Cartridges	40 g SepaFlash® E Series silica cartridge (Order number: S-8101-0040) 40g SepaFlash® E Series neutral alumina cartridge (Order number: S-8601-0040-N)	
Sample loading	110 mg, dry loading method	
Wavelength	220 nm	
Mobile phase	A: n-hexane B: ethyl acetate	
Flow rate	30 ml/min	
Eluting gradient	% B	Time / (min)
	5	0
	50	30

Results and Discussion

According to the preparative chromatogram, these two alkaline heterocyclic compounds were barely eluted on the SepaFlash® silica cartridge, which may be a result of irreversible adsorption of the sample to the silica stationary phase (as shown in Figure 2). In comparison, apparent peaks were observed in the chromatogram of SepaFlash® neutral alumina cartridge, suggesting appropriate retention time and good resolution was obtained for the two components of the sample mixture while neutral alumina cartridge was utilized (as shown in Figure 3).

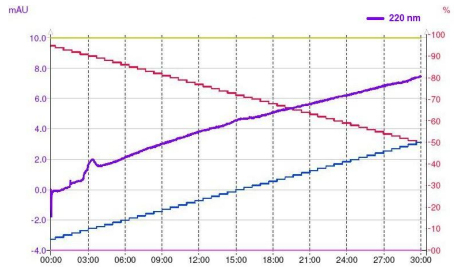

Figure 2. The chromatogram of the sample in SepaFlash® silica cartridge.

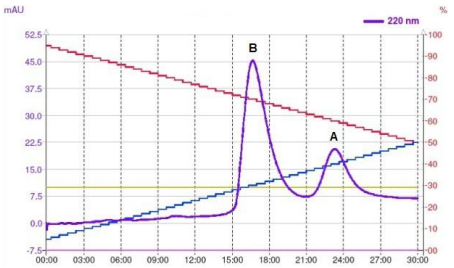

Figure 3. The chromatogram of the sample in SepaFlash® neutral alumina cartridge.

The alkaline compounds usually show severe tailing in chromatogram or even irreversible adsorption while eluted in silica cartridges. The reason for this may be attributed to the interaction between the sample and the silicone groups on the silica gel surface. As contrast, the above mentioned samples show good peak shape and high recovery rate while eluted in neutral alumina cartridges, which barely have hydroxyl groups interacting with acidic or alkaline compounds. Moreover, some samples are unstable in acidic conditions.

Handling such kind of samples with silica cartridges may result in sample deterioration since the silica gel has weak acidity. However, such embarrassing situation could be avoided if neutral alumina cartridges were employed.

In summary, SepaFlash® neutral alumina cartridges are a considerable solution for the separation and purification of alkaline compounds or acid-sensitive samples.

About SepaFlash® E Series alumina cartridges

SepaFlash® E Series alumina cartridges include a series of cartridges pre-packed in different cartridge sizes (as shown in Table 2 and Figure 4).

Table 2. SepaFlash® E Series alumina cartridges

Item Number	Column Size	Flow Rate (mL/min)	Max.Pressure (psi/bar)
S-8601-0004-N	8 g	10-30	300/20.7
S-8601-0012-N	24 g	15-45	300/20.7
S-8601-0025-N	50 g	15-45	300/20.7
S-8601-0040-N	80 g	20-50	300/20.7
S-8601-0080-N	160 g	30-70	200/13.8
S-8601-0120-N	240 g	40-80	200/13.8
S-8601-0220-N	440 g	50-120	150/10.3
S-8601-0330-N	660 g	50-120	150/10.3
S-8601-0800-N	1600 g	100-200	100/6.9
S-8601-1600-N	3200 g	150-300	100/6.9
S-8601-3000-N	6400 g	150-300	100/6.9

Figure 4. SepaFlash® E Series alumina cartridges.

**Note 1: The item numbers in the above table represent neutral alumina cartridges.*
For acidic alumina cartridges, the last letter N should be replaced with A.
For alkaline alumina cartridges, the last letter N should be replaced with B.
Note 2: pH range for acidic alumina is 3.8 – 4.8; 6.5 -7.5 for neutral alumina; and 9.0 – 10.0 for alkaline alumina.

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