Technical Note 20668

Assessment of the Stability of 4 µm Solid Core Particles for the Analysis of Non-Steroidal Anti-Inflammatory Drugs

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Key Words

Accucore XL, column stability, fused core, superficially porous, solid core, ibuprofen

Abstract

The analysis of non-steroidal, anti-inflammatory drugs on a 4 µm solid core C8 HPLC column is described. A method was created using isocratic conditions for separation to demonstrate column stability. The column was proven to remain stable following 2,200 injections and 45,000 column volumes of a test mixture containing naproxen, fenoprofen, and ibuprofen.

Introduction

Thermo Scientific™ Accucore™ XL HPLC columns use Core Enhanced Technology™ to facilitate fast and highly efficient separations. The 4 µm diameter particles are not totally porous, but instead have a solid core and a porous outer layer. The optimized phase bonding creates a series of high coverage, robust phases. The tightly controlled 4 µm diameter of Accucore particles results in lower backpressures than typically seen with fully porous particles of the same diameter. The nature of the Accucore XL particles provide an improvement in performance of an analysis compared to that seen on columns using fully porous 3 µm and 5 µm particles.

Stability of a column is of critical importance in obtaining high quality data. Deterioration in column performance can result in batch and validation failures that can effect timelines and result in loss of both time and money. Therefore, a great deal of value is placed on showing that columns remain reproducible when being used for extended periods of time. An analytical method was developed to enable the assessment of Accucore XL HPLC column stability.

Experimental Details

Sample Preparation

Primary standards of theophylline, naproxen, fenoprofen, and ibuprofen were prepared separately in methanol at a concentration of 1000 µg/mL. A working standard was prepared by preparing a 10 mL solution of Ibuprofen, fenoprofen, theophylline, and naproxen by adding 1 mL ibuprofen, 0.5 mL fenoprofen, 0.5 mL theophylline, and 0.025 mL naproxen primary standards to 1.975 mL acetonitrile and 6 mL 20 mM ammonium formate, pH 3.

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Separation Conditions		Part Number
Instrumentation:	Thermo Scientific Dionex™ UltiMate™ 3000 HPLC system	
Column:	Accucore XL C8 4 µm, 50 x 2.1 mm	74204-052130
Mobile phase:	Acetonitrile / 20 mM ammonium formate, pH 3 (40:60 v/v)	
Backpressure:	50 bar	
Column temperature:	30 °C	
Injection volume:	2 µL (partial loop)	
Flow rate:	0.3 mL/min	
UV detection:	233 nm (data rate 20 Hz)	

Results

A stable analytical HPLC method was developed to assess the robustness of the Accucore XL HPLC column. Using isocratic conditions, full separation of the non-steroidal, anti-inflammatory drugs (NSAIDs) naproxen, fenoprofen, and ibuprofen was performed. The Accucore XL C8 HPLC column was shown to be stable for the entire experiment with no indication of deterioration in chromatography even after 2,200 injections, which equates to 45,000 column volumes.

The reproducibility of the Accucore XL HPLC column for the chromatographic separation of the NSAIDs is summarized in Table 1. It is evident that the data for all analytes is matched with excellent precision with the % RSD retention factor of ≤0.80% and % RSD normalized efficiency ≤1.52%. A typical chromatogram of the NSAIDs is seen in Figure 1.

	Naproxen		Fenoprofen		Ibuprofen	
	k'	$N/N_{(n1-5)}$	k'	$N/N_{(n1-5)}$	k'	$N/N_{(n1-5)}$
Mean	2.26	0.986	4.80	0.996	7.99	1.002
% RSD	0.80	1.52	0.77	0.66	0.77	0.36

Table 1: Assessment of the stability on the Accucore XL C8 HPLC column assessed for 2,200 injections. Efficiency was assessed by normalizing efficiency values to the mean of the efficiencies for the first five samples injected.

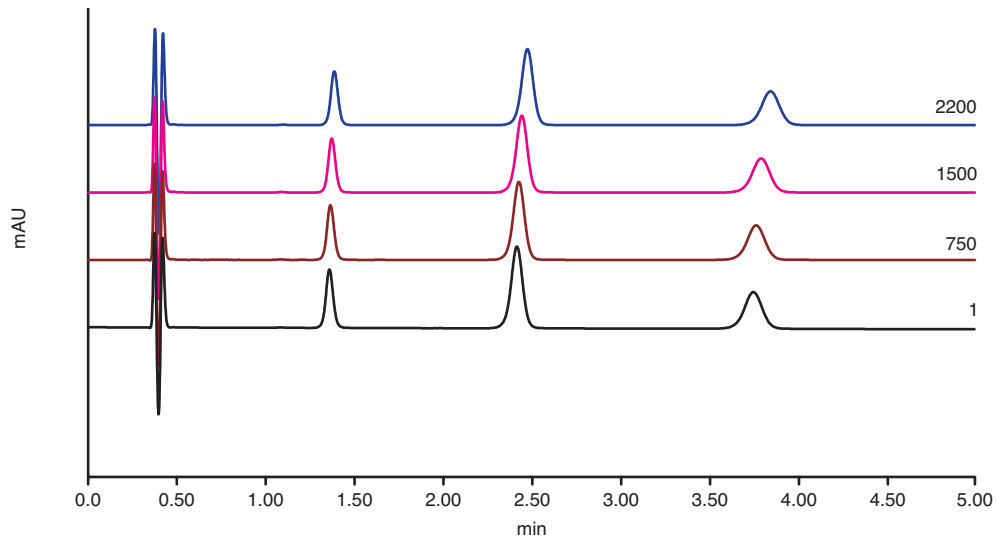

Figure 1: Separation of naproxen, fenoprofen, and ibuprofen, with theophylline as a t0 marker. Injections 1, 750, 1500, and 2,200 are shown.

Figures 2 and 3 are illustrations of the stability data. It is apparent there was a degree of variability within the middle of the analysis, brought about by instrument related issues. However, there is no indication of instability of the Accucore XL HPLC column, even after 2,200 injections, as both the retention and efficiency remained consistent to the end of the analysis.

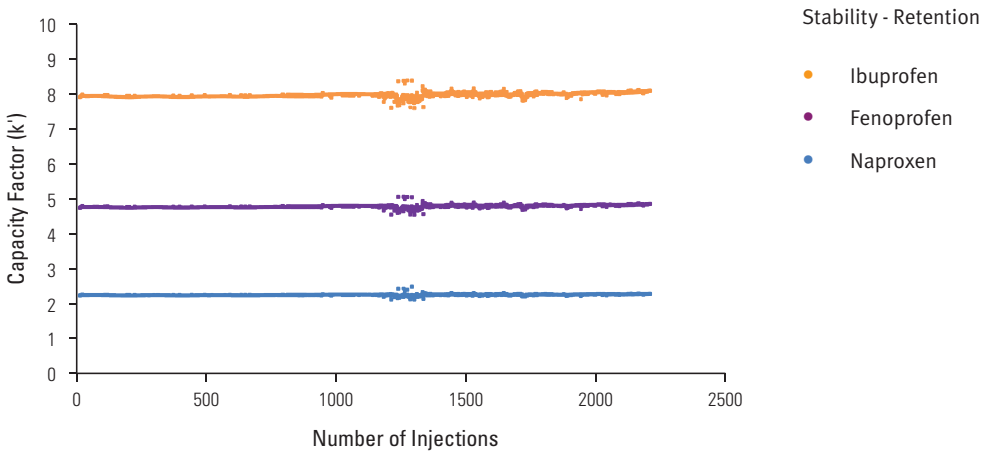

Figure 2: Assessment of the reproducibility of retention for naproxen, fenoprofen, and ibuprofen over 2,200 injections on the Accucore XL C8 HPLC column

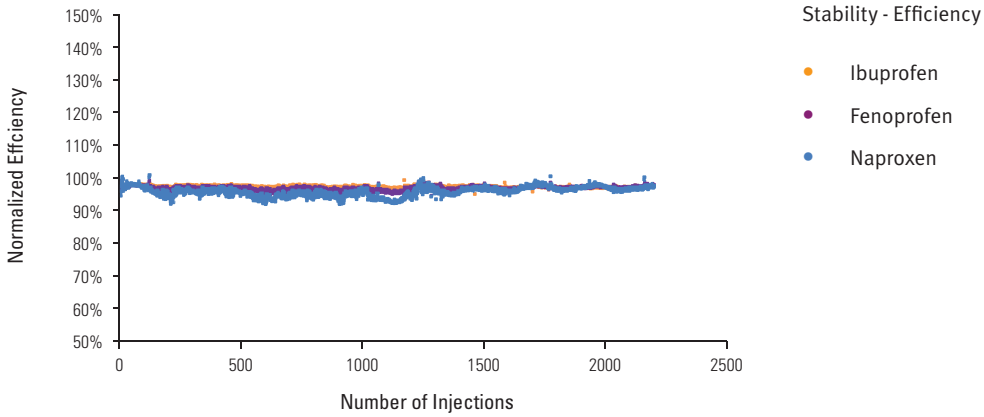

Figure 3: Assessment of the reproducibility of efficiency for naproxen, fenoprofen, and ibuprofen over 2,200 injections on the Accucore XL C8 HPLC column

Conclusion

It has been demonstrated that the Accucore XL C8 HPLC column is stable following 2,200 injections of a solution containing the NSAIDS naproxen, fenoprofen, and ibuprofen. Using isocratic conditions, full separation of the NSAIDs was achieved. The precision of capacity factors and efficiency for the NSAIDs over the course of the investigation is excellent, and the Accucore XL C8 HPLC column was shown to provide excellent peak shape for these compounds.

The Accucore XL HPLC columns have been proven to remain stable over the course of the analysis, which makes them ideal columns to choose to obtain high-performing and robust HPLC separations.

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TN20668_E 01/13S