Analysis of Non-Steroidal Anti-Inflammatory Drugs Using a Highly Pure, High Surface Area C18 HPLC Column

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Application Note 20673

Key Words

Syncronis C18, diclofenac, naproxen, aspirin, ibuprofen, sulindac, piroxicam, fenoprofen, NSAIDs, non-steroidal anti-inflammatory drugs

Abstract

This application note demonstrates the use of the Thermo Scientific™ Syncronis™ C18 HPLC column for the analysis of non-steroidal anti-inflammatory drugs. Syncronis C18 columns provide a fast simple method with good hydrophobic retention, excellent peak shape and high resolution.

Introduction

One of the key goals for the chromatographer is to achieve a consistent, reproducible separation. The selection of a highly reproducible HPLC column is essential if this goal is to be attained. The Syncronis column range has been engineered to provide exceptional reproducibility due to its highly pure, high surface area silica, dense bonding and double endcapping, all controlled and characterized through the use of rigorous testing.

Non-steroidal Anti-Inflammatory Drugs (NSAIDs) are medications used in reducing inflammation, relieve pain (analgesic) and to lower temperature (fever). NSAIDs are the most prescribed medications for treating conditions such as pain, arthritis, fever and migraine.[1,2] Separating non-steroidal anti-inflammatory drugs with good resolution can be problematic in liquid chromatography. In this application the Syncronis C18 phase was employed to achieve the separation of seven most commonly used NSAIDs.

Experimental Details

Consumables	
Fisher Scientific™ HPLC grade water	W/0106/17
Fisher Scientific HPLC grade acetonitrile	A/0626/17
Fisher Scientific HPLC grade methanol	M/4056/17
Thermo Scientific Autosampler vial kit	A4954-010

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Separation Conditions		Part Number
Instrumentation:	Syncronis C18, 3 µm, 100 x 3.0 mm Thermo Scientific HPLC system	97103-103030
Column Temperature:	30 °C	
Injection volume:	10 µL	
Flow rate:	0.60 mL/min	
UV detection:	260 nm	
Mobile phase A:	0.1% formic acid in water	
Mobile phase B:	0.1% formic acid in acetonitrile	
Gradient:	50-75% B in 5 minutes	
Run time:	7 minutes	
Pressure:	207 bar	
Wash solvent:	90:10 (v/v) water: acetonitrile	

Sample Preparation

Stock solutions of diclofenac, naproxen, aspirin, ibuprofen, sulindac and fenoprofen were prepared in methanol at a concentration of 1.0mg/mL. Piroxicam was prepared in a methanol:water (1:1 v/v) solution at 1.0mg/mL. The stock solutions were mixed and diluted with water to yield a final working solution containing each compound at the following concentration:

Compound	Concentration (µg/mL)
Naproxen	15
Ibuprofen	100
Diclofenac	30
Aspirin	30
Sulindac	30
Piroxicam	30
Fenoprofen	30

Results

Analysis was carried out on a 3 µm Syncronis C18 column as illustrated in Figure 1. The Syncronis column showed excellent resolving power and provided a very simple method for the separation of all seven compounds. The high surface area silica, dense bonding and double endcapping assist in minimizing secondary silanol interactions and as a result Syncronis columns provide good peak shapes and high resolving power. The reproducibility of the method was assessed by doing six consecutive runs. The method was shown to be very robust with consistent retention times. The chromatograms are shown overlaid in Figure 2. The relative standard deviation of the retention from the six consecutive runs was calculated to be less than 0.12% for all seven compounds. (Table 1)

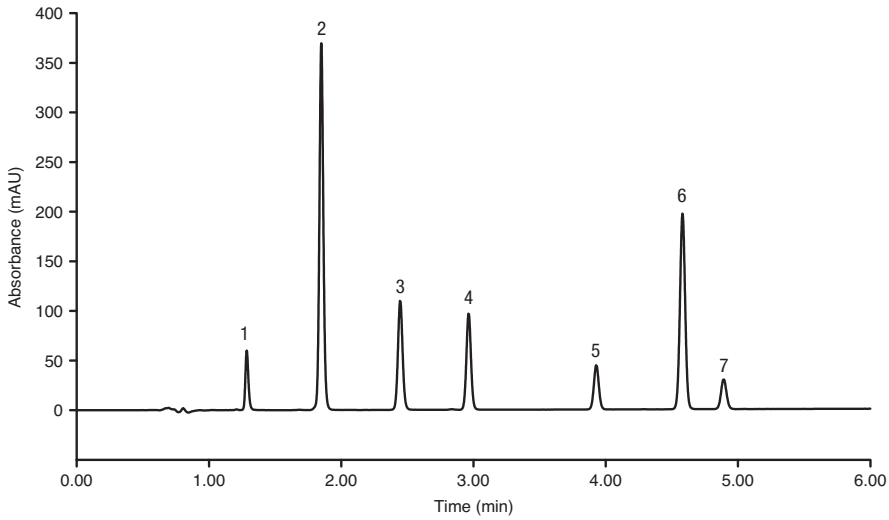

Figure 1: Separation of non-steroidal anti-inflammatory drugs

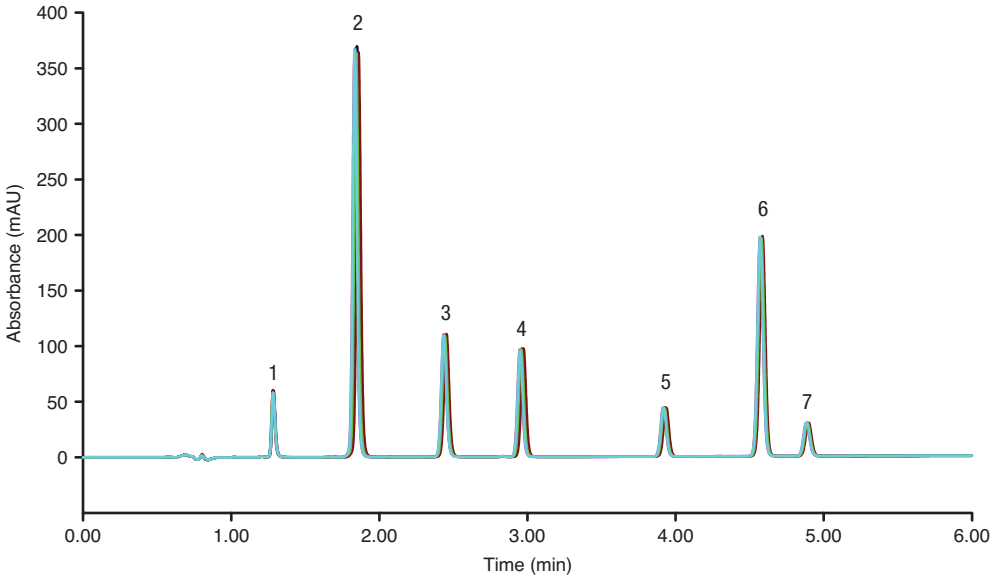

Figure 2: Overlaid chromatograms showing method robustness

Peak ID	Retention Time (min)	Asymmetry (EP)	Resolution(USP)	T_R RSD (%) n=6
1	1.28	1.21	12.05	0.03
2	1.86	1.08	9.97	0.11
3	2.45	1.08	8.13	0.06
4	2.97	1.04	14.52	0.07
5	3.94	1.02	9.19	0.04
6	4.59	1.02	4.18	0.03
7	4.90	1.05	n.a.	0.02

Table 1: Results summary

Conclusion

The Syncronis C18 column successfully separated all seven non-steroidal anti-inflammatory drugs with a relatively short retention time and a very simple method. This application demonstrates that Syncronis C18 columns

- retain and resolve non-steroidal anti-inflammatory drugs
- give excellent peak shape
- provide excellent resolution
- give consistent and highly reproducible methods

The method overall proved to be robust, simple and highly reproducible.

References

1. www.medicinenet.com
2. Wikipedia, http://en.wikipedia.org/wiki/NSAIDS

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AN20673_E 11/12S