

SEPARATION OF NAPROXEN USING NEW IMMOBILIZED COLUMN – CHIRALPAK® IH

APPLICATION NOTE

INTRODUCTION

DAICEL Corporation has been at the forefront of chiral stationary phase (CSP) development for over 30 years. In 2002, the coated CSP AS-H (5 µm) and AS-3 (3 µm) were commercialized, and since then have been widely utilized by chromatographers because of their unique separation characteristics. During the last 10 years, DAICEL has introduced a range of immobilized chiral columns to circumvent the issue that all coated columns are incompatible with most organic solvents. Now, DAICEL has added two new immobilized alternatives, CHIRALPAK® IH (5 µm) and IH-3 (3 µm), to its line of existing CSPs. The IH phase is an immobilized version of CHIRALPAK® AS, which is an amylose polymer derivatized with α-methyl benzyl carbamate moieties.

Unlike AS-H, IH can be used under screening conditions that use both DMF and THF. As shown in Figure 1, prolonged exposure to DMF at 40°C on a 0.46 cm i.d. x 15cm long IH-3 column does not affect the column's separation ability on a standard trans-stilbene oxide QC test.

In addition to this important improvement, testing has also shown that IH and IH-3 can achieve baseline separation on some compounds that AS-H or AS-3 cannot, such as 1-(1-Naphthyl)ethanol (Figure 2).

EXPERIMENTAL

Naproxen, a commonly used NSAID, was subjected to optimized normal phase conditions on both AS-H and IH to compare selectivity and resolution, as well as productivity in a preparative method.

DISCUSSION

Where AS-H was previously unable to separate the two enantiomers of naproxen, IH does so with good selectivity and resolution (Figure 3).

Under preparative conditions, because Naproxen has better solubility in Ethyl Acetate compared to Isopropanol, a nearly 120x increase in productivity is observed (Figure 4). N.B. Ethyl Acetate is not compatible with coated CSPs.

FIGURE 1: PROLONGED EXPOSURE TO DMF

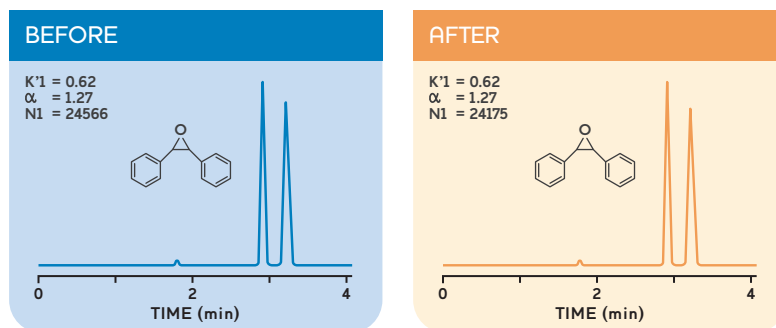
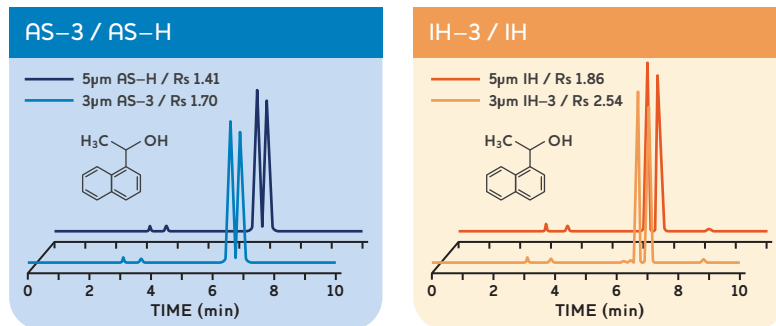


FIGURE 2: 1-(1-NAPHTHYL)ETHANOL BASELINE SEPARATION



CHROMATOGRAPHIC CONDITIONS

Column: Daicel CHIRALPAK AS-H
Column Size: 4.6 mm i.d. x 250 mm long
Mobile Phase: n-Hex/EtOH/TFA (90/10/0.1) v/v/v
Flow rate: 1.0 mL/min.
Temperature: 25° C
UV: 254 nm

CHROMATOGRAPHIC CONDITIONS

Column: Daicel CHIRALPAK IH
Column Size: 4.6 mm i.d. x 250 mm long
Mobile Phase: n-Hex/EA/TFA (80/20/0.1) v/v/v
Flow rate: 1.0 mL/min.
Temperature: 25° C
UV: 254 nm

FIGURE 3: SEPARATION OF TWO NAPROXEN ENANTIOMERS

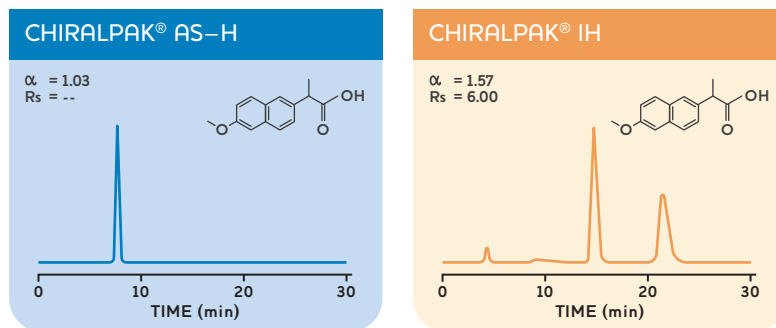
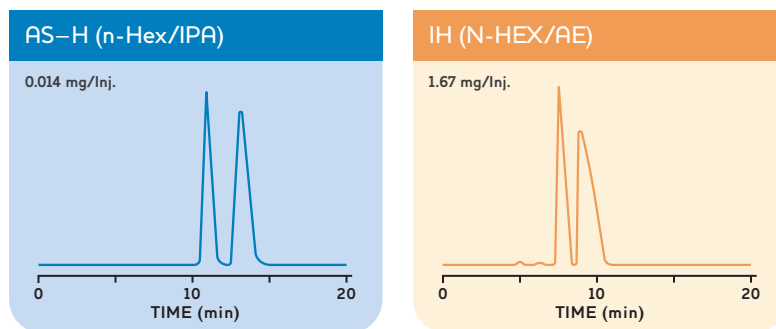


FIGURE 4: INCREASE IN PRODUCTIVITY



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