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FULL SHORT COMMUNICATION

Elevated Temperature Separations on Hybrid Stationary Phases with Low Proportions of Organic Modifier in the Eluent

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Abstract The properties of two thermally stable organic/inorganic hybrid columns, XTerra MS C_{18} and XTerra phenyl were examined at elevated temperatures, using a number of test compounds, including acetophenone derivatives and phenols, with 5 and 10% methanol in water as the mobile phases. The thermodynamics of the separations were examined. The study demonstrated that a low proportion of organic modifier and moderate temperatures gave similar separations to higher temperature with superheated water, potentially reducing stress on the column and analytes.

Keywords High temperature liquid chromatography · Hybrid stationary phases · Van't Hoff relationships · Methylene selectivity

Introduction

In recent years there has been a growing interest in high-column temperature separations in HPLC in the range 60–200 °C [1–6]. These conditions offer a number of advantages: retention times are considerably reduced giving faster separations, the eluent viscosity is reduced enabling smaller particle sizes to be employed thereby increasing column efficiency, and for suitable analytes it is possible to use superheated water alone as the eluent as an environmentally friendly eluent and eliminating the detection interferences from the organic component of conventional eluents [7–11].

However, although water is environmentally friendly, cheap and non-hazardous, even at 200 °C it is only a moderately strong solvent and is often unable to rapidly elute relatively non-polar organic compounds, such as steroids [12] which can lead to long-retention times. This has lead to an interest in the use of low proportion of organic modifier in the eluent. In an early study Pawlowski and Poole [13] suggest that the presence of a small proportion of organic modifier (1% acetonitrile) may be desirable for solvation and peaks shapes on ODS-bonded silica columns but would have little effect on the solvation characteristics of the column. A secondary advantage of using a low percentage of organic phase is that it avoids the dewetting/phase collapse that can occur when using C18 bonded silicas with purely aqueous phases [14–16].

In high-temperature separations, the thermal and chemical stability of the stationary phase is a major consideration, particularly for silica-based stationary phases. Traditional ODS-bonded silica column are frequently unstable and have short life-time above 60–80 °C. Alternative column materials, such as zirconia-based phases, polystyrene-divinyl benzene columns, and porous graphitic carbon have been compared in a number of studies [for example 17–19]. More recently, organic-silica hybrids, such as methyl linked XTerra and ethylene linked XBridge phases have become available and have been found to work well under conventional organic-water and superheated water conditions to 150–220 °C [18–21]. In addition, these materials have become available as sub 2-μm stationary phases, which have also been employed successfully for high-temperature separations [11, 22, 23].

This study examines the use of temperature with low proportions of organic modifier in the eluent for the

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