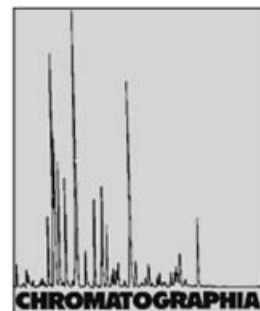


Thermal Stability of Thiazide and Related Diuretics During Superheated Water Chromatography



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Abstract

The separations of eleven thiazide and related sulfonamide diuretics using superheated water chromatography at up to 200 °C on an XBridge C18 column were compared with conventional liquid chromatography at ambient temperature. Most of the sulfonamide diuretics were thermally stable, but many of the thiazides were degraded. The most sensitive were the dihydrothiazides, including cyclopenthiiazide and hydrochlorothiazide, which were converted to the corresponding aminobenzendisulfonamides in the pre-column heater. The *N*-methyl thiazides, including methyclothiazide, appeared to be more stable but degraded on the column and of the thiazides, chlorothiazide eluted largely unchanged although some on-column degradation was observed for benzthiazide. The results confirmed that in superheated water chromatography problems may be encountered with analytes with low storage stability at ambient temperature.

Keywords

Superheated water chromatography
Hybrid silica column
XBridge C18 column
Thiazide diuretics

Introduction

In recent years there has been an increasing interest in the application of high temperature in LC as a variable in method development and in particular as a way of reducing retention times or

limiting or eliminating the need for organic solvents in the mobile phase [1–8]. However, the widespread adoption of the environmentally friendly superheated water chromatography (also known as subcritical or pressurised hot water chromatography) has been limited

because of concerns about the stability of the stationary phases and of analytes at the temperatures that are frequently employed, often up to 200 °C. However, previous studies have shown that employing polymeric, metal oxides such as zirconia, and most recently hybrid stationary phases can provide columns, which are sufficient stable for extended use [1, 2, 7, 10, 11].

A range of different analyte groups have been studied using superheated water chromatography, from hydrocarbons, vitamins, numerous pharmaceuticals to steroids, and including functional groups which might be expected to be hydrolysed or oxidised, such as esters or phenols. There have been few reports of analyte loss or degradation other than in labile analytes, such as aspirin [12] and thiamine [13], which have limited aqueous stability at ambient temperature. However, although Edge and co-workers were able to separate paracetamol and aminobenzoic acids at 60 °C they were degraded at 180 °C [6]. In a drug metabolite study this group also reported [9] that there was a loss of a diglucuronide during isothermal separations at 124 °C but this analyte could be detected using an isobaric temperature gradient. One reason for the general analyte stability is probably that in most cases the exposure of the analyte to the