



Fast Separation of Nitrosamines

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Interest in the analysis of nitrosamine compounds is increasing as researchers become more aware of their toxicity and presence in the environment, foods, and pharmaceuticals. The extraordinary chemical and thermal stability of zirconia-based stationary phases allows for the rapid separation of these polar, basic compounds with good peak shape and efficiency. This note shows the separation of nine low molecular weight nitrosamines using a DiamondBond™-C18 column.

Introduction

N-nitrosamines are present in both food and the environment, and have been shown to be highly carcinogenic, with toxic levels as low as micrograms per kilogram. The nitrites and nitrates commonly used as preservatives in food can cause the reaction of secondary amines to form N-nitrosamines (1). Detection of these N-nitrosamines has been demonstrated using both UV detection at 230 nm (2) and mass spectrometry (3) coupled to liquid chromatography, however these separations are typically quite lengthy, ranging from 15-35 minutes.

The excellent chromatographic selectivity and thermal stability of zirconia-based phases allows much faster separation of these compounds at either high or low pH, where the electrospray ionization of the positively charged amines for detection by mass spectrometry is facilitated.

Experimental

A standard mixture of N-nitrosamines obtained from Supelco contained the following nine compounds: dimethylnitrosamine, ethylmethylnitrosamine, diethylnitrosamine, dipropylnitrosamine, dibutylnitrosamine, diphenylnitrosamine, nitrosomorpholine, nitrosopiperidine, nitrosopyrrolidine. The mixture was separated at 75 °C using a DiamondBond-C18 column using the following chromatographic conditions:

Column: 4.6 mm x 100 mm DiamondBond™-C18
Mobile Phase: 2.5-90% B from 1-3 minutes
A = 10mM Ammonium hydroxide, pH 9.5
B = ACN
Flow rate: 4.0 ml/min.
Injection Vol.: 1.0 µl
Detection: UV at 230 nm

Even at a modest temperature of 75 °C the separation is rather fast, with full resolution of all nine compounds in just over 3 minutes.

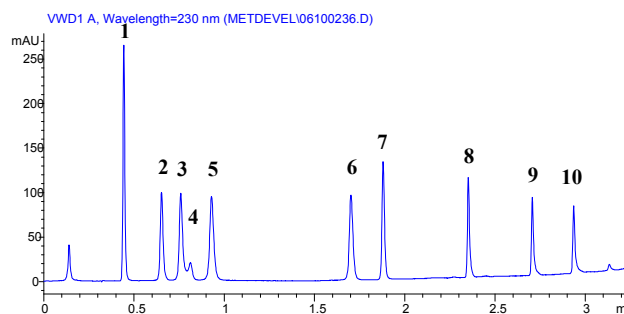


Figure 1: Separation of nitrosamines at 75 °C.

We note that our analysis shows ten peaks for the nine compound mixture. We believe peak number four in this separation may be an impurity in the mixture.

Note that even temperature-sensitive compounds can benefit from modest increases in temperature, making faster analysis possible. ZirChrom's technical support group has extensive experience in this area, and would be happy to help you with your particular application.

ZirChrom columns combine high efficiency with improved stability for extraordinary separations.

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References

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