



B116 Using Coupled Columns With Ultra High-Performance Supercritical Fluid Chromatography (UHPSFC) for the Analysis of Synthetic Cathinones

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After attending this presentation, attendees will understand the uses and principles of UHPSFC with coupled columns for the separation of synthetic cathinones.

This presentation will impact the forensic science community by demonstrating the benefits of coupled column UHPSFC in drug analysis. Screening or confirmation of bath salts, including their positional isomers, using coupled columns is accomplished with significantly greater reliability compared to the use of a single column. Identification can be accomplished by retention time, Ultraviolet (UV) spectrum, and molecular ion and major fragment ions.

UHPSFC has been used to separate bath salts, including their positional isomers. The current method uses a Torus™ DIOL column with a 10mM ammonium formate in methanol, run isocratically with 3% methanol and 97% carbon dioxide as the mobile phase. This method separates 10 out of 15 controlled bath salts and 28 out of 34 isomers with a resolution of 1 or greater. In order to achieve better separation, coupled columns using different stationary phases were investigated. The two coupled columns were connected through a short length of narrow bore tubing and isocratic conditions were employed to the combined columns. The retention times from each column were approximately additive, so an estimation of the retention times of the coupled columns could be achieved prior to testing.¹ In order to investigate if greater separation of bath salts and their positional isomers could be achieved by using a coupled column approach, eight columns were tested. The columns include seven 1.7µm 3.0x100mm achiral columns (Torus™ DIOL, CSH Fluoro-Phenyl, HSS C18 SB 1.8 µm, BEH 2EP, 1-AA, Torus™ DEA, Torus™ 2PIC) and one 2.5µm 3.0x150mm chiral column (Trefoil CEL 1).

A combination of Torus™ DIOL and Torus™ 2PIC run with 3% 10mM ammonium formate in methanol has demonstrated significantly better resolution, in which 11 out of 15 controlled bath salts and, more importantly, 32 out of 34 positional isomers were separated in less than 15 minutes. In contrast to the use of a single column, six positional isomers of pentedrone and 4-methylethcathinone are now fully resolved. Percent methanol, temperature, pressure, and flow rate can also be adjusted in order to increase the resolution.

The effects of using coupled columns compared to single columns on the retention factor, selectivity factor, and efficiency were also investigated. During coupling, both the connecting tubing and temperature gradients caused by the increased pressure can contribute to band broadening.

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Reference(s):

1. Phinney Karen W., Sander Lane C., Wise, Stephen A. Coupled achiral/chiral column techniques in subcritical fluid chromatography for the separation of chiral and nonchiral compounds. *Anal Chem.* 1998, 70, 2331-2335.

Synthetic Cathinone, Supercritical Fluid, Coupled Column

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