B127 Applicability of Ultra High-Performance Supercritical Fluid Chromatography (UHPSFC) as a Separation Technique for Synthetic Cannabinoids and Synthetic Cathinones

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After attending this presentation, attendees will understand some separation principles of UHPSFC, in particular the applicability to the separation of synthetic cannabinoids and synthetic cathinones. Attendees will gain insight into whether UHPSFC should be used by forensic chemists in their general analysis scheme for emerging drugs.

This presentation will impact the forensic science community by presenting a technique that has the potential to be the best chromatographic method for distinguishing between very similar solutes, such as positional isomers and diastereomers. This information could be particularly valuable in aiding the analyst in determining which controlled substance is present and to distinguish between a controlled and non-controlled emerging drug.

The recently introduced separation technique UHPSFC produces highly efficient and rapid separations performed on a new generation of analytical SFC instruments with an environmentally friendly mobile phase, containing carbon dioxide as the major component. Supercritical and subcritical carbon dioxide has properties that are intermediate between a liquid and a gas, giving it excellent diffusivity while maintaining liquid-like properties. UHPSFC, similar to high-performance liquid chromatography and ultra high-performance liquid chromatography, is advantageous for drugs that are thermally labile, polar and non-volatile solutes that are problematic for Gas Chromatography (GC) analysis. UHPSFC offers increased selectivity for very similar compounds due to interactions with the stationary phase such as hydrogen bonding, dipole and pi-pi interactions, and is particularly useful for the separation of designer drugs including synthetic cannabinoids and synthetic cathinones ("bath salts").

The Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG), which is responsible for setting standards for drug analysis, does not list SFC as an approved separation technique. In order to be listed for SWGDRUG under Category B, a separation technique should not only offer reasonable separation ability but be orthogonal to accepted techniques such as GC and Liquid Chromatography (LC).

In these studies, four achiral columns, including 2-PIC, Diol, DEA, and 1-AA (1.7µm 3.0 x 100m), and three chiral columns, including AM1, CEL1, and CEL2 (2.5µm 3.0 x 150mm), were investigated for the separation of synthetic cannabinoids and bath salts using carbon dioxide with various modifiers and additives. The modifiers included methanol, acetonitrile, ethanol, and isopropanol, while the additives included ammonium formate and ammonia. Detection was carried out by photo diode array-Ultraviolet (UV) in series with single quad Mass Spectrometry (MS). The synthetic cannabinoids studied consisted of 24 controlled drugs, including a pair of positional isomers and two pairs of diastereomers, nine non-controlled positional isomers of controlled JWH-018, a non-controlled positional isomer of controlled JWH-073, and a non-controlled diastereisomer of HU-210. The "bath salts" investigated included 15 controlled drugs, with three pairs of positional isomers, seven non-controlled positional isomers of controlled pentedrone and 4-methylcathinone, four non-controlled positional isomers of controlled mephedrone and buphedrone, three non-controlled positional isomers of controlled α-PVP, two non-controlled positional isomers of controlled 4-MEPP and α-PBP, and one non-controlled positional isomer of controlled methcathinone, butylone, methylone, pentylone, and MDPV.

Although significant co-elution was observed for the controlled synthetic cannabinoids using UHPSFC, the co-eluting compounds could easily be distinguished by MS detection. Furthermore, the work demonstrates that UHPSFC is particularly valuable for the separation of isomers (both controlled and non-controlled) and all the positional and most diastereomers of the synthetic cannabinoids were resolved using this group's optimized parameters in less than ten minutes. For the "bath salts," good overall resolution was obtained for all compounds including their positional isomers in less than eight minutes using optimized UHPSFC conditions. All of the controlled "bath salts" could be distinguished by a combination of retention time and MS detection.

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Lastly, UHPSFC separations for the above emerging drugs were compared to separations obtained by both GC and Ultra High-Pressure Liquid Chromatography (UHPLC), with particular attention paid to the separation of positional isomers and diastereomers. The latter techniques can be particularly problematic for the separation of positional isomers.[1] The degree of orthogonality of UHPSFC, GC, and UHPLC was demonstrated for the separation of both synthetic cannabinoids and bath salts using principal component analysis.

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Reference(s):

1. Marginean I., Rowe W.F., Lurie I.S. The role of ultra high performance liquid chromatography with time of flight detection of the identification of synthetic cannabinoids in seized drugs, *Forensic Sci. Int*. 249 (2015) 83-91.

Synthetic Cannabinoids, Synthetic Cathinones, Supercritical Fluid

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