



K35 A Rapid and Comprehensive Analysis of HFAA Derivatized “Bath Salts,” Synthetic Cathinones, and Amphetamines in Postmortem Blood by Supported Liquid Extraction (SLE) With Gas Chromatography-Mass Spectrometry Detection

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After attending this presentation, attendees will understand new trends in “Bath Salts,” synthetic cathinones, and amphetamine abuse. Attendees will learn a unique approach to utilizing Supported Liquid Extraction (SLE), the challenges of derivatizing with Heptafluorobutyric Acid Anhydride (HFAA), and techniques to overcome analytical column contamination due to byproducts caused from HFAA derivitization.

This presentation will impact the forensic science and law enforcement communities by highlighting the abuse patterns for new designer drugs. Abuse of synthetic cathinones and amphetamines, particularly in our youth, is on the rise. A comprehensive approach to extraction, detection, and quantification of “Baths Salts,” synthetic cathinones, and amphetamines is required to fully assess the extent of this problem. Challenges related to identifying these compounds, including compound volatility and derivitization, will be discussed. The method presented suggests one possible approach that may be utilized to reliably characterize and identify these new compounds. Efficient testing procedures are a necessary first step in a comprehensive surveillance program.

3,4-Methylenedioxypyrovalerone (MDPV), is a new “designer” drug with observed toxicities including tachycardia, hypertension (vasoconstriction), insomnia, hyperthermia, mydriasis, panic attack seizures, and aggressive behavior. MDPV is a psychoactive, synthetic analog of the CNS stimulants cathinone and pyrovalerone. Because of their appearance, MDPV is often referred to as “bath salts.”

A new procedure to facilitate a rapid, comprehensive, and simultaneous detection and analysis of amphetamine, phentermine, methamphetamine, amantadine, nicotine, pseudoephedrine, methylenedioxymphetamine (MDA), methylenedioxymethamphetamine (MDMA), phenylpropanolamine (PPA), MDPV, mephedrone, and 3,4-Methylenedioxy-N-ethylamphetamine (MDEA) was developed using Supported Liquid Extraction (SLE). Beta-Phenethylamine was also analyzed as it is a common byproduct which is often observed in postmortem blood specimens.

Ten postmortem samples that tested positive for the amphetamine drug class via ELISA screen were further evaluated using this new method. Extraction is rapid, utilizing a small sample volume and involving two steps. The two step sample preparation and extraction utilizes an ISOLUTE SLE cartridge (Biotage, Charlotte, NC) combined with a positive-pressure manifold. The SLE cartridge composition contains Diatomaceous earth, a porous structure. Using SLE, the aqueous sample solution that has been pH adjusted with alkali or NH₄OH will penetrate into the pore of the Diatomaceous earth. When eluted with a hydrophobic solvent, an extraction interface occurs between the two liquid phases. The high surface area of the Diatomaceous earth provides for enhanced extraction efficiency while alleviating much of the technical expertise associated with traditional Liquid-Liquid Extraction (LLE). SLE is able to minimize the impact from hemolyzed specimens, a common concern when dealing with postmortem blood samples. In addition, costly disposal of organic waste is not required because of minimal extraction volumes. This extraction is followed by derivitization with HFAA and Gas Chromatography-Mass Spectrometry (GC/MS) detection. This method was challenged by analytical column contamination from the HFAA which was overcome by incorporating a post conversion/derivitization clean up procedure that uses a phosphate buffer. This clean up procedure helped to maintain chromatographic quality without adding a labor intensive step.

With this methodology, a single analytical protocol can be used to conduct the confirmation test for a comprehensive panel of sympathomimetic amines on postmortem blood samples that preliminarily tested positive by immunoassay for amphetamines. Criteria for method validation which include accuracy, inter-assay precision, intra-assay precision, and analytical measurement range studies will be reported. In comparison with traditional LLE, this method offers considerable savings in sample volume, extraction time, solvent usage, waste production and disposal.

Bath Salts, Postmortem, Supported Liquid Extraction (SLE)