K75 Comparison of Solid Phase Extraction (SPE) and Supported Liquid Extraction (SLE) Columns for the Extraction of 23 Novel Psychoactive Substances From Blood and Urine

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After attending this presentation, attendees will be able to compare SLE and SPE columns and identify the correct choice for optimum recovery of cathinones and NBOMes from a variety of biological matrices.

This presentation will impact the forensic science community by increasing awareness of extraction options for the determination of novel psychoactive substances in forensic toxicological samples.

SLE columns are now commercially available and pose an alternative to SPE. SLE has commercial benefits over SPE in that it does not produce the same amount of solvent waste and can be carried out in fewer steps depending on the type of SPE column being compared.

Recently, there has been an influx of new synthetic substances to the recreational drug market with laboratories struggling to keep pace in this "cat and mouse" game. To improve detection rates, it is vital that laboratories are using the optimum sample preparation technique to allow maximum analyte recovery and sample throughput.

The goal of this research was to determine whether SLE+ columns can be used as a possible alternative sample extraction method for the detection of synthetic cathinones and NBOMes and to evaluate which clean-up method produces the maximum recovery across a range of 25 drugs.

Blank methanol, urine and blood samples (1mL) were spiked with 100µL of 10µg/mL solutions of various different NPS's (methiopropamine, flephedrone, mephedrone, MDPV, 2-DPMP,butylone, ethylone, naphyrone, 5-APB, 6-APB, 3-MeO-PCE, methoxetamine, benzedrone, 25B-NBOMe , 25C-NBOMe , 25D-NBOMe , 25E-NBOMe , 25H-NBOMe, 25I-NBOMe, Mescaline-NBOMe, 25N-NBOMe, 25P-NBOMe, 25T2-NBOMe, 25T4-NBOMe, and 25T7-NBOMe). Urine samples were pH adjusted to 10.8 using 1% ammonium hydroxide (NH_4OH). To each sample prepared for SPE, 1mL of 0.1M phosphate buffer (pH6) was added before centrifugation for ten minutes at 4,000rpm. UCT's ZDSAU020 columns were conditioned using methanol, deionized water, and phosphate buffer before loading samples. Columns were washed using deionized water, 0.1M acetic acid, and methanol. Samples were eluted using methylene chloride; iso-propanol; NH_4OH (78:20:2). For SLE, following pH adjustment with 1% ammonium hydroxide, samples were loaded directly to Biotage's® SLE+ columns. The sample was held on the column for five minutes before being eluted with 2x4mL of ethyl acetate. Internal standards (mephedrone-D_3, methylone-D_3, ethylone-D_5, MDPV-D_8, and 25I-NBOMe-D_3) were added to the collection tubes prior to elution. Post-extraction, samples were evaporated using a stream of nitrogen, derivatized using 50µL of PFPA:ethyl acetate at 70oC for 40 minutes, before being evaporated again and reconstituted in 100µL of ethyl acetate. Samples were analyzed by GC/MS with the SLE and SPE results being compared directly to unextracted methanolic standards at the same concentration.

All drugs were successfully extracted from each matrix using both SPE and SLE columns. For blood, SLE+ columns provided a higher recovery rate of drug than the SPE columns, with an average increase of 10% (recovery ranging from -47% to 80%). SPE-extracted urine samples more efficiently providing an average of 5% increase in recovery rates (recovery ranging from 47% to 92%).

In conclusion, when analyzing blood samples, SLE+ should be used whereas SPE is more efficient for the extraction of these analytes from urine.

SPE, SLE, Novel Psychoactive Substances