K77 Qualitative Analysis of Designer Stimulants and Bath Salts Chemicals in Blood, Serum/Plasma, and Urine by LCTOF

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After attending this presentation, attendees will be able to discuss the use of Liquid Chromatography Time-Of-Flight Mass Spectrometry (LC/TOF/MS) for the screening of designer stimulants and hallucinogens in biological samples and identify considerations that reduce the risk of false positive findings.

This presentation will impact the forensic science community by increasing forensic toxicologists' understanding of the strengths and limitations of LC/TOF analysis and the need for confirmatory testing by independent methods.

LC/TOF is becoming an increasingly popular technique for drug screening in forensic and clinical toxicology laboratories. The technique is based on high-efficiency liquid chromatographic separations, coupled with a detection system that confirms the identity of the analyte by assessing its retention time and accurate mass relative to an analytical standard and its accurate mass. A panel was developed and validated for the detection of 40 analytes, including popular designer drugs such as MDPV, mephedrone, methylone, ethylone, naphyrone, 5-MeODALT, 5-MeO-DIPT, 2C-D, 2C-E, 2C-H, 2C-I, 2C-T-2, 2C-T-7,and others. The compounds were selected based on the fact that they are scheduled either at the state or federal level and are increasingly being found in "Bath Salts" type of products for purposes of abuse.

Samples (0.5mL) were buffered with 0.1M borax buffer followed by liquid-liquid extraction using 70:30 n-butylchloride:ethyl acetate. The acquisition method used a run time of 10 min with a flow of 0.700mL/min. The method used six representative deuterated internal standards to monitor analyte recovery. A target cut-off of 10ng/mL was selected for most compounds in blood and urine. Criteria used to evaluate positivity were retention time, mass accuracy, and percent concentration compared to the cutoff.

Validation of the assay consisted of assessment of precision around the cut-off, stored sample stability (room temperature (light and dark), refrigerated and frozen), carryover, autosampler stability, interference, sensitivity, and specificity.

The validation of this panel yielded an overall sensitivity of 99.7% with a range of 72% to 100% and selectivity of 99.0% with a range of 79% to 100%. Generally, analyte stability was shown to be sufficient in refrigerated and frozen samples and insufficient otherwise, whether light protected or not. Naphyrone in serum was an exception, which was shown to be unstable after one day. No carryover was detected following a sample spiked at 100 times the cutoff. Potential interferences were investigated by testing various mixed analyte pools, and did not yield any false positive findings.

The method's reproducibility was demonstrated by observing internal standard area, performance of the positive and negative controls, and the limited number of false positives. False positives only appeared to be an issue with isobaric pairs of analytes, such as 3-FMC and flephedrone, and MDMA and mephedrone. For this reason, when either member in the pair is present, confirmation testing will be done for both.

LC/TOF is a useful tool for forensic toxicology screening; however, the limitations of the technique must be acknowledged. Recognizing isobaric compounds is key to using LC/TOF as a screening tool, since the molecular mass of the analytes is the main identifying criteria. The success of the technique is highly demanding of high resolution chromatography and chromatographic quality and stability during the assay. Mass accuracy is one parameter that can help in determining the positivity of an analyte. Ion suppression due to sample matrix is another issue that must be addressed. Using representative deuterated internal standards is one way of determining the likelihood of ion suppression in a given sample.

LC/TOF, Designer Drugs, Stimulants

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