B22 Method Validation and Detection of Adulterants in 1,031 Seized Drug Exhibits by High Resolution Mass Spectrometry

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Learning Overview: After attending this presentation, attendees will be able to describe current trends in the number and variety of adulterants encountered in seized material and implement an efficient workflow for the analysis of drugs of abuse and adulterants in seized material on a high-resolution mass spectrometry platform.

Impact on the Forensic Science Community: This presentation will impact the forensic science community by providing information about a new and rapid methodology to screen for a wide range of cutting agents in seized materials. Also, attendees will have information about the prevalence of adulterants, many of which have significant toxicity, in seized drug evidence from several United States state jurisdictions.

Cutting agents, classified as diluents (pharmacologically inactive; e.g., sugars) and adulterants (pharmacologically active; e.g., phenacetin), are commonly used to increase profits. Cutting agents themselves as well as mixtures and ratios are constantly changing over time, which can increase risks to the user's health as a result of their side effects. Most laboratories in the United States report only controlled substances on the Drug Enforcement Administration (DEA) list (Schedules I to IV) or per their state regulations, which leads to under-reporting of other substances (adulterants) that may contribute to the adverse effect profile of illicit drug use. The goal of this work was to develop and validate a screening method to analyze 54 adulterants and scheduled drugs in seized drug evidence.

Analysis was performed on a VANQUISH™ Ultra High-Performance Liquid Chromatography (UHPLC) coupled to a Q-Exactive™ Orbitrap™/Mass Spectrometry (MS). The UHPLC was operated using a reverse-phase gradient using 0.1% formic acid in ultrapure water (MPA series) and 0.1% formic acid in acetonitrile (MPB series) for chromatographic separation on an Accucore™ C18+ analytical column (100mm x 2.1mm, 1.5µm). The UHPLC gradient initial conditions of 95A:5B changing to 5A:95B (3mins, hold till 3.5mins) and return to initial conditions and hold for 2.5mins at a flow rate of 0.3mL/min for a total run time of six minutes. Precursor ions were acquired by full MS scan (50–750m/z) via positive electrospray ionization. Precursor isolation was performed using dd-Ms^2. Fragmentation was achieved using stepped collision energy of 20, 40, and 80eV. Data processing was performed using Trace Finder™ software with an Extracted Ion Chromatogram (XIC) list containing 54 compounds, including fragments and accurate mass library.

A fit-for-purpose validation was performed for this method including precision, limit-of-detection, and carryover experiments following United Nations Office on Drugs and Crime (UNODC) and Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) guidelines. The method was successfully validated for 52 substances. Gabapentin and carisoprodol failed to be properly detected and identified following the processing parameters due to coelution with theophylline, codeine, and alprazolam. Adjustments in the processing criteria rules were made in case of detection of one or more compounds mentioned above in the same sample. The method was free from carryover up to the concentration of 1mg/mL. The limit of detection was defined for all compounds at 100ng/mL.

De-identified evaporated autosampler vials from seized drug exhibits collected in Pennsylvania, North Carolina, Texas, Virginia, New York, Georgia, and Maryland were received and linked to an in-house identifying number, date of receipt by the originating lab, and the zip code or county of origin. Prior to analysis, the samples were reconstituted in 1,000µL of mobile phase. The following data is representative of 1,031 samples. Overall, 51 substances were detected in the samples, and the most prevalent toxic adulterants found were lidocaine (12.97%), levamisole (10.84%), quinine/quinidine (10.45%), caffeine (10.16%), and procaine (8.71%). Fentanyl and heroin were mostly adulterated with quinine/quinidine and lidocaine, while cocaine was mostly adulterated with levamisole (56.2% of the samples) and phenacetin (28.4% of the samples). Tetrahydrocannabinol (THC) and methamphetamine were less adulterated in general, only 7.5% and 15.8 of those samples contained adulterants, respectively, while fentanyl, heroin, and cocaine samples were adulterated 89.3%, 73.2%, and 93.2% of the time, respectively.

Knowledge about cutting agents is commonly overlooked either because they are not detected or not reported. This leads to a lack of information that could be useful for management of acute intoxications in hospitals or criminal investigations or in helping in the identification of routes of trafficking. Common reporting practices, however, frequently do not provide information about the prevalence of these toxic adulterants.

Seized Drugs, Cutting Agents, Toxicity

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