B168 Comprehensive and Definitive Characterization of Drug Microcrystals

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After attending this presentation, attendees will learn the extent to which microcrystalline tests can aid in the identification of drugs of abuse. With it now possible to obtain structural data using XRD of the known microcrystals, drug samples can be identified using simpler analysis such as IR and Raman with the aid of the reliable libraries.

Based on the findings of the study, the scope for furthering microcrystalline tests in modern forensic laboratory has been highlighted. With a link between the drug sample and the microcrystal structure formed after reagent addition now recognized, this presentation will impact the forensic community and/or humanity by demonstrating how analysis such as mass spectrometry may no longer be necessary, with the growth of libraries in the future making analysis more efficient.

The aim of this study is to create a database of instrumental and visual characterization of microcrystals used in forensic analysis. The study utilized five reagents ($PtCl_6$, $AuCl_4$, $AgNO_3$, K_2CdI_4 and PbI_2- KOAc.), two of which are used in ASTM standard methods for microcrystal identification. The analysis and characterization of crystals was conducted using polarizing light microscopy, image recognition, infrared and Raman microspectroscopy, and X-Ray diffraction.

Central to this work was the definitive identification of crystal structures via the X-Ray diffraction studies. Previous work in here has shown the value of this approach as applied to GHB and related compounds. Here, work focused on much smaller crystals, specifically a group of related phenylethylamines (amphetamine, methamphetamine, phentermine, and ephedrine). Crystals were observed with several of the reagents and similarities and differences were noted. Structures of both the individual ion-pair unit structures and the aggregated crystal lattices were obtained. Most of the structures were hydrated and showed reproducible spatial arrangements. Variations in structures correlated as expected with differences in the heavy metal atom (Cd, Au, Pt, etc.). For example, amphetamine with gold chloride formed two independent molecular structural units within the crystal lattice, $[NH_3ChMeCH_2Ph]\bullet AuCl_4$ with two water molecules. These units then organized themselves into a reproducible orthorhombic crystal.

These same crystals were also characterized using micro-FTIR using a diamond ATR cell and Raman microspectrometry at two wavelengths. Because these spectra were obtained from crystals of known structures, reliable libraries can be created. Thus, forensic practitioners do not need X-Ray diffraction data to identify microcrystals but can rely now, in many cases, on secondary measurements such as IR, Raman, and micrographic measurements, to definitively associate a crystal structure with presence of a specific drug. In effect, a microcrystal test can be considered to be a chemical extraction technique. Thus, having a means of linking observed structure to identification moves microcrystal from presumptive and subjective to the realm of objective and definitive testing and significantly increases their value.

Microcrystals, X-Ray Diffraction, Phenylethylamines

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