## B124 Single Crystal X-Ray Diffraction in Forensic Drug Analysis

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After attending this presentation, attendees will better understand the crystal structure of the precipitate resulting from the gold chloride microcrystal test with ecgonine, several phenethylamines, and 3,4-methylenedioxypyrovalerone (MDPV). Additionally, attendees will understand the crystal structure of several cathinones ("bath salts") and the additional information that X-ray diffraction can provide to the analyst. This presentation will describe how these structures were determined using single crystal X-ray diffraction techniques and the potential implications for the microcrystal testing of other drugs.

This presentation will impact the forensic science community by providing essential information regarding the molecular structure and interactions within the crystal lattice of the compounds investigated as well as the benefits of single crystal X-ray structure determination.

The analysis of controlled dangerous substances by microcrystal tests has been largely empirical, relying on the experience and training of the analyst to make conclusions regarding the identity of microcrystalline precipitates viewed microscopically. The microscopist had little support beyond the optical characteristics provided by the polarized light microscope to base his/her conclusions. While the morphology and optical properties of the resulting crystals have been studied and documented, very little structural information has been available on the molecular level. Single crystal X-ray diffraction is an ideal technique for determining the chemical composition of crystals containing highly diffractive heavy metals, such as gold chloride complexes.[1]

Ecgonine precipitates as crystals with the gold chloride reagent as both the anhydrous salt and the hydrate. Single crystals of ecgonine-gold chloride was grown for comparison to previous work with cocaine-gold chloride.[2] For the hydrate, the asymmetric unit consists of four protonated cations of ecgonine, surrounded by five gold chloride anions and seven waters, one of which is a hydronium ion for charge balance. The crystal lattice contains a network of water molecules woven between the cations and anions making hydrogen bonds with the hydroxyl and carboxyl functions of the ecgonine molecules. The anhydrous crystal of the ecgonine salt demonstrates close contact bonding between the gold chloride anions and the ecgonine cations and an intramolecular hydrogen bond between the protonated nitrogen and the carboxyl oxygen.

A series of related phenethylamines (ephedrine, amphetamine, and methamphetamine) were grown with gold chloride as single crystals for X-ray diffraction. Both ephedrine and methamphetamine precipitated as salts with the 1:1 ratio of cation-to-anion as seen with other microcrystal tests. The ephedrine-gold chloride complex shows a stratified internal arrangement in which the phenyl rings of the ephedrine cations are in bands every one-third of the unit cell and the gold chloride anions reside in clusters near the protonated N atoms of the cations.

However, amphetamine produced a unique cycloaurate complex with a centrally located Au atom bound bidentate to the amphetamine ligand and to two Cl atoms. In the methamphetamine-gold chloride crystal structure, the gold chloride anion is aligned between the phenyl rings of the cations, deviating from a line drawn between the centroids of the phenyl rings by less than 0.6Å.

The "bath salt" MDPV was studied as the gold chloride precipitate. A single hydrogen bond exists from the protonated N atom of the cation to the proximal anion. Additionally, several law enforcement seizures of crystal MDPV were examined directly by X-ray crystallography as submitted to the laboratory. The analysis of these samples has provided some insight into the nature of these dangerous compounds.

The purpose of this research is to gain an understanding of how the structure of the selected compounds and their gold (III) chloride complexes may play a role in the formation of crystals visualized by the traditional microcrystal test. This is an ongoing research project at Rutgers University. Further crystallographic studies will focus on other related drug compounds and additional microcrystal tests.

**Reference(s):**

1. McCrone W.C., 1992: Microcrystal tests and the *Frye* rule. *Microscope*. 40(3): 198
2. Wood M.R. et al., 2007: The gold(III) tetrachloride salt of L-cocaine. *Acta Cryst*. C 63: m33-m35

**Drug Analysis, Crystallography, Microcrystal Tests**





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