The Detection of Date Rape Drug Residues Using X-Ray Diffraction

Emily A. Walsh
Forensic Science Department
Dr. Virginia M. Maxwell

Abstract

A predominant problem investigators and forensic scientists encounter is difficulty in the detection of date rape drugs in a drug facilitated sexual assault (DFSA) victim's system, more specifically their blood, hair, and urine. After a matter of hours these fast acting drugs have little chance of being detected with modern toxicological techniques due to their rapid metabolism by the body. Proving the use of these drugs can be very hard to establish due to the challenge of detection. This project utilized X-ray diffraction (XRD) in order to detect and identify date rape drugs on various materials. The date rape drugs used for the purposes of this project were Gamma-Hydroxybutyric acid (GHB), Chloral Hydrate, Ketamine, Flunitrazepam (Rohypnol), and MDMA (Ecstasy). XRD has been implemented in many forensic science laboratories due to low cost, versatility, and the non-destructive nature of x-ray diffraction. The focus of this research was to evaluate the use of XRD for low

used in the field of forensic science for the purpose of analyzing solid material evidence from minute trace amounts to large complex mixtures with several different compositions. The method has great versatility because crystalline structure is the only requirement for analysis. Materials composed of metallic, organic, and inorganic compounds can be analyzed using this technique.(4) XRD is non-destructive to the materials being analyzed, which

between parallel lattice planes, and d the Bragg angle. More specifically, the Bragg angle is the angle between the incident beam and a lattice plane. (6)

Figure 1: Diagram of Bragg's Law (6)

The principle behind X-ray diffraction involves the crystallinity of a sample scattering-rays. The x-rays produced by the instrument are passed through the sample and the ordered arrangement of atoms scatter the x-rays. The constructive interference created by the scattered beams moving in phase with each other ultimately produces a diffracted beam. (7) The ability to distinguish one sample from another comes from each compound having a characteristic crystal structure, resulting in distinct peak positions. This unique diffraction pattern is directly influenced by the geometry of the crystal lattice. (6) Diffraction patterns are influenced by both the intensity and angle of the diffracted beams gathered by the detector component of the instrument. (8) When the sample diffracts the x-rays, that are then detected by the instrument, the angle at which they were diffracted is what produces unique diffraction peak patterns.

Methods and Materials

The instrument used for this project was the Rigaku MiniFlex II Desktop X-ray Diffractometer. The software utilized for analyzing the obtained spectra was MDI Jade 9.

A standard silicone test was performed to ensure the instrument was working properly every day before samples were analyzed. Preliminary samples of the following were run to gain familiarity with the instrument: a business card, two brands of baby powder, two brands of duct tape, milk thistle, aspirin, ibuprofen, acetaminophen

Figure 2: The X-ray Diffractometer used for this project.

and analyzed using XRD with the same parameters and peak tables were created using Jade.

A mixture of 5 mL ethanol and 0.0028 g Ketamine was prepared in a 10 mL falcon tube. A few drops of the mixture was pipetted onto a flat zero background holder. This was left out to evaporate. Once a residue was formed, analysis with XRD with the same parameters was carried out and peak tables obtained with Jade.

Results

Fabrics analyzed with XRD give diffraction patterns of only a few (3 to 5) peaks because of the uniformity in the structure of the material. This minimal diffraction pattern is shown by the denim (cotton) spectrum in Figure 3.

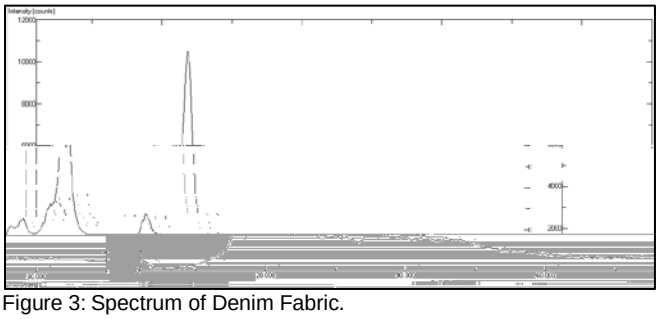

Figure 3: Spectrum of Denim Fabric.

Each of the date rape drugs as well as the confectionary sugar sample analyzed with XRD yielded clear diffraction patterns with numerous peaks as shown in Figures 4 and 5. These background spectra were utilized in later peak comparison and assignment of peaks in the mixture samples.

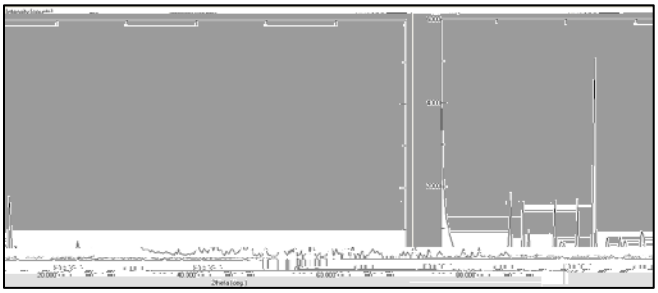

Figure 4: Spectrum of Ketamine.

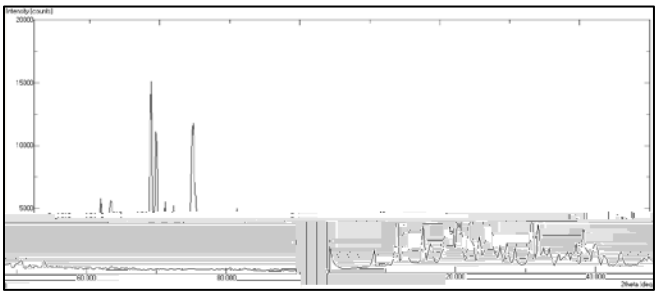

Figure 5: Spectrum of Confectionary Sugar.

The dry mixture samples and the paste samples transferred onto the fabrics yielded more complex spectra due to the multiple components of the samples. Examples of these spectra are shown in Figures 6 and 7. Despite this complexity, XRD had the power to analyze these multifaceted mixtures resulting in being able to distinguish between the individual component diffraction patterns. Coupled with the Jade software, XRD was able to provide enough data to assign to the individual peaks to the component in which it originated for all the mixture samples. The dry 10% drug mixtures yielded peaks that could be attributed to the confectionary sugar, but more importantly the peaks for the constituent drugs were unchanged and visible. The same could be concluded for the paste on the fabrics.

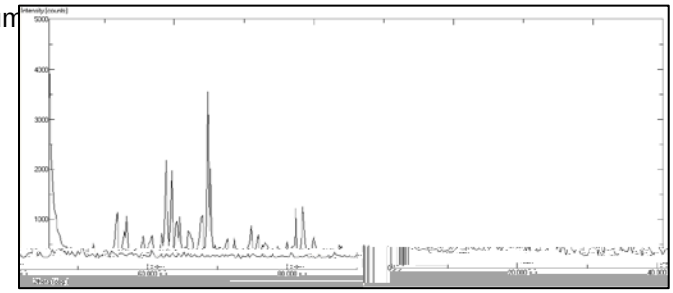

Figure 6: Spectrum of 10% Mixture of Ketamine and Confectionary Sugar.

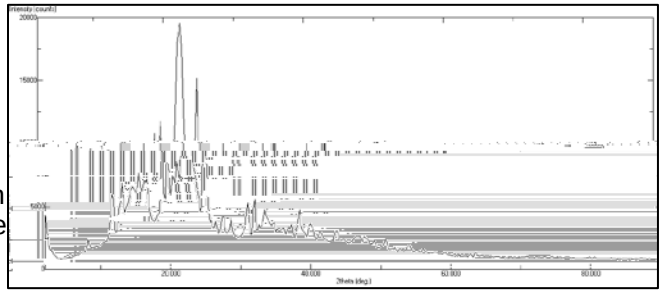

Figure: 7: Spectrum of Denim Fabric with 10% Ketamine and Confectionary Sugar Paste.

When the residue, seen in Figure 8, from the evaporation of the Ketamine and ethanol mixture was completed, consequently leaving a crystal structure on the zero background holder, the XRD was able to analyze the residue. Through visual comparison of the diffraction pattern as seen in Figure 9 with the pattern seen in Figure 4, Ketamine was concluded to be present in the sample.

Figure 8: