K36 NMR Analysis of 3,4-methylenedioxy-N- methylamphetamine (MDMA or Ecstasy) and its Metabolites in Urine

Elise Champeil, PhD, John Jay College of Criminal Justice, 445 West 59th Street, New York, NY 10019; Gloria Proni, PhD, John Jay College of Criminal Justice, Science Department, 445 West 59th Street, New York, NY 10019; and Jonathan Liu, MSc, John Jay College, 445 West 59th Street, New York, NY, 10019*

After attending the presentation, attendees will learn about the use of NMR spectroscopy for the detection of drugs of abuse in urine. Real case studies are also presented.

This presentation will impact the forensic science community by introducing a new technique to detect and quantify the presence of MDMA (ecstasy) in human urine.

Drug testing in urine is a common technique used today. Current methods of testing urine for drugs and their metabolites include HPLC, GC-MS, or immunoassay analysis. These methods all have their drawbacks.[1] Recently, nuclear magnetic resonance, (NMR), has emerged as a means of analyzing drugs and drug metabolites in urine. There is literature precedence describing the use of NMR spectroscopy to identify compounds in urine from intoxication.[2] There are many benefits to using NMR spectroscopy: NMR is non-destructive and samples can be analyzed as many times as desired. There is also little sample preparation required.

3,4-methylenedioxy-*N*-methylamphetamine, more commonly called MDMA or "ecstasy", is a synthetic drug similar in structure to methamphetamine.

In this project, we investigated the practicality of using NMR spectroscopy to detect and quantify the presence of 3,4-methylenedioxy- *N*-methylamphetamine (MDMA or ecstasy) in human urine.

First, a calibration curve was established with spiked samples of real urine. To determine the standard deviation, seven independent urine samples spiked with the different compounds at a concentration of 0.05mg/mL were run. Variance (S^2) and standard deviation (S) of the measurements were calculated.

As for the LOD, the very nature of NMR makes it impossible to determine as it depends on the amount of scans used for the experiment. In this study the experimental time was limited to overnight experiments, allowing a quantification in the 0.01 mg/mL concentrations range.

Following this, real urine samples from MDMA users were analyzed. The real samples were collected following an IRB approved protocol. Five different samples were collected. This presentation discusses the spectra of the urine obtained from these 5 volunteers. Figure 1 shows the spectrum of the first sample. Surimposed in gray is the spectrum of MDMA spiked urine (0.50 mg/mL). All peaks for the protons of MDMA are clearly visible.

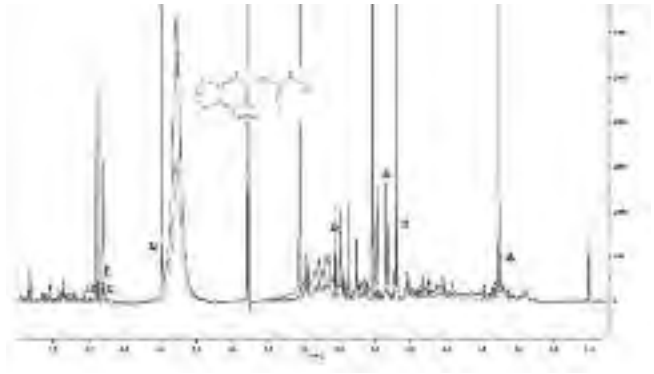
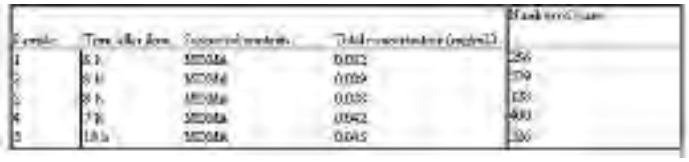


Figure 1: Sample 1, 256 scans. Superimposed in gray is the spectrum of MDMA spiked urine (0.50 mg/mL).

The results are summarized in the following table:



 ** Presenting Author*

These results suggest the 1H NMR spectroscopy could provide a convenient tool for the rapid detection of MDMA in human urine. This method presents the advantage of a rapid diagnosis with little of urine needed and no sample preparation. Furthermore, samples were analyzed within 20-30 minutes. The NMR method should be useful in rapidly confirming the diagnosis of poisoning.

The limitation of using NMR for the identification of MDMA is that at lower concentrations, the presence of small amounts of metabolites or other therapeutic agents can interfere. In that case, the quantification procedure can be difficult.

References:

[1] Smith, M. (2006). *Principles of ForensicToxicology,* Washington, D.C.: AACC Press. (pp. 119-139).

[2] Imbenotte, M., Azaroual, N., Cartigny, B., Vermeersch, G., Lhermitte, M. (2003). *Forensic Sci Int, 133*, 132-135. Komoroski, E. M., Komoroski, R. A., Valentine, J. L., Pearce, J. M., Kearns, G. L. (2000). *J Anal Toxicol, 24*, 180-187. Meshitsuka, S., Inoue, M., Seki, A., Koeda, T., Takeshita, K. (1999). *Clin Chim Acta, 279*, 47-54. Savin, S., Cartigny, B., Azaroual, N., Humbert, L., Imbenotte, M., Tsouria, D., Vermeersch, G., Lhermitte, M. (2003). *J Anal Toxicol, 27*, 156-161. Wahl, A., Azaroual, N., Imbenotte, M., Mathieu, D., Forzy, G., Cartigny, B., Vermeersch, G., Lhermitte, M. (1998). *Toxicology, 128*(1), 73-81.

NMR, Ecstasy, Urine

 ** Presenting Author*