A213 Rapid Screening of Synthetic Cannabinoids With NMR and DART®-MS

Ling Huang, PhD, Michael A. Marino, BS, and Mercurio Veltri, PharmD, Hofstra Univ, Chemistry Dept, Hempstead, NY 11549; and Robert B. Cody, PhD, and John Dane, PhD, 11 Dearborn Rd, Peabody, MA 01960*

After attending this presentation, attendees will understand the fundamental principles of Direct Analysis At Real-Time Mass Spectrometry (DART®-MS) and Nuclear Magnetic Resonance (NMR) spectroscopy, specifically their applications for rapid detection of synthetic cannabinoids in herbal incenses. Attendees also will appreciate the power of combining two spectroscopic methods to promptly elucidate accurate structures of cannabinoids with no ambiguity.

This presentation will impact the forensic science community by introducing a combined spectroscopic approach to quickly solve the challenges in identifying synthetic cannabinoids and isomers found in herbal incenses or potpourri, also known as “spice” or “fake pot.”

The recently-passed S.3187 included five classes of synthetic cannabinoids into Schedule I controlled substance list. It creates great challenges for forensic scientists to rapidly screen these “cannabimimetic agents” in numerous forms of herbal incenses due to the similarity of isomer or analog structures. Newer compounds are being synthesized promptly to circumvent the ban, which exacerbates the analytical difficulties for forensic labs, some of which are already experiencing backlogs.

Our hypothesis is to combine the rapid speed of DART®-MS with H1 NMR to rapidly screen and concretely confirm the identities of synthetic cannabinoids in herbal products with minimum sample preparation. The mass spectra provide molecular weights as well as fragment information. NMR spectra confirm the structure with no ambiguity. The whole process takes less than two hours. Novel isomers and analogs can be quickly identified and structures confirmed, sometimes without the presence of standards.

As suggested in previous literature, DART® is an atmospheric ionization technique that can be used to instantly ionize illicit drugs sprayed herbal base materials with no sample preparation. The limitation, however, is the difficulty of DART-MS to elucidate structural isomers. With the help of NMR, the isomeric or analog structures can be confirmed.

A commercially available high resolution DART®-TOF mass spectrometer system was used for the direct analysis of all samples. The DART® was operated in positive ion mode using helium gas at a gas heater temperature of 300° C. The powder samples were introduced into the DART® gas stream as a coating on the outside of a glass rod. For the herbal samples, three random pieces of plant material were selected from a given sample bag and then held in the DART® gas stream with forceps. The CAD experiments were done by varying the mass spectrometer cone voltage at 0.2sec intervals to induce fragmentation. For the NMR study, a simple methanol (3mL) extraction was employed to remove synthetic cannabinoids from 50mg of solid herbs, before paper filtration, solvent evaporation and redissolving in NMR solvent such as $CDCl_3$ or d-6 acetone. After one-hour H1 NMR scan, the chemical shift values from synthetic components were detected and used to confirm DART®-MS finding. The chemical shift values of signature peaks were analyzed to identify synthetic cannabinoids. Our study suggests that most signature peaks from naphthoyl and benzoyl indoles are in 7-8.5ppm or 4-4.2ppm ranges, which do not interfere with the herbal background signals from blank herbal extracts. A flow chart was developed to quickly identify the cannabinoids based on their signature chemical shift values.

With the combination of DART®-MS and NMR, 30 herbal samples and seven powder samples from distributors were quickly screened for synthetic cannabinoids. Three of the powder samples were found to be mislabeled and the other three contaminated with other cannabinoid(s). Synthetic cannabinoids were found in all but one herbal incense. Among the herbal samples investigated, AM-2201, JWH-122, JWH-210, and RCS-4 were found to be the predominant ingredients.

In conclusion, accurate identification of synthetic cannabinoids in powder and herbal samples can be rapidly achieved with DART®-MS along with NMR confirmation. The simple NMR method can differentiate isomers such as JWH-019, JWH-180, and JWH-122. These combined spectroscopic methods can help forensic chemists to achieve accurate identification within two hours which increases the analytical throughput and helps to decrease sample backlog.

Cannabinoids, NMR, DART-MS

 ** Presenting Author*