K61 Weeding Analytes Out of Marijuana: The Identification and Quantification of Pesticides in Cannabis Utilizing Comprehensive Gas Chromatography

Emily Ly, and Frank Dorman, PhD, 107 Whitmore Labs, University Park, PA 16802; and Jack Cochran, BS, and Julie Kowalski, BS, 110 Benner Cir, Bellefonte, PA*

After attending this presentation, attendees will understand the principles of analyzing and quantifying cannabis for specific cannabinoids such as Cannabinol (CBN), Cannabidiol (CBD), Cannabichromene (CBC), cannbigerol (CBG), and Delta-9-Tetrahydrocannabinol (THC) using Gas Chromatography with Flame Ionization Detection (GC/FID). They will also understand the concepts and reasons for testing for pesticides contained on cannabis utilizing Gas Chromatography/Mass Spectrometry (GC/MS) and Gas Chromatography/Electron-Capture Detectors (GC/ECD) as well as the practical applications for such analyses. Finally, attendees will understand how Comprehensive Gas Chromatography (GCxGC) can be utilized to potentially determine both potency as well as trace organics characterization in a single analysis.

This presentation will impact the forensic science community in a variety of different ways, but loosely falls into two classes: characterization of cannabis as a potential pharmaceutical; and, potentially fingerprinting the trace compounds in cannabis to determine the point of origin. Testing for potency can help determine the identity and abundance of target cannabinoids that have therapeutic qualities. These qualities have been confirmed to relieve pain, control nausea, stimulate appetite, and decrease ocular pressure.[1] With this knowledge, medical marijuana can be grown more effectively by lessening the main psychoactive component, THC, which may cause discomfort in patients, and increasing the target therapeutic cannabinoids.[2] The abundance of THC and other cannabinoids is affected by a variety of factors including environmental conditions, harvesting periods, and the sex of the plant.[3] Furthermore, many pesticides, fungicides, and insecticides are used to treat the cannabis plant. This is of concern for any person that consumes the material due to the residual toxins that are potentially harmful. Moreover, a study was conducted in 1992 for the United States Drug Enforcement Administration (DEA) which determined that chemical profiles of cannabis samples could be used to locate the geographical origin.[4] However, the system could only eliminate possible sources of origin and therefore had low specificity due to the fact that only cannabinoid constituents were analyzed.[4] By identifying and quantifying the pesticides on the cannabis plant, it may be possible to develop a “chemical fingerprint” relating to compounds used by growers to increase the crop yield. This information may allow law enforcement agencies determine and/or link the source location of the confiscated illicit drug.

One hundred and six different samples of illicit marijuana were analyzed. These were obtained directly from local law enforcement personnel. Samples were initially homogenized, and the finely ground marijuana, weighing approximately 0.2 to 2g, was mixed with 10mL of acetonitrile and 10mL of water in 50mL centrifuge tubes, similar to the QuEChERS extraction procedure developed at the USDA.[5] Water was added to increase the extraction efficiency of more polar pesticides.[6] This solution was spiked with internal standards and pesticides for recovery purposes before soaking for an hour. The solution was shaken for 30 min with a vortex mixer. QuEChERS EN salts were added and the solution was shaken for 1 min.[5] This was followed by a 5 min phase separation utilizing centrifugation.[5] The supernatant was removed and refrigerated.[6] QuEChERS extraction is an efficient method that minimizes organic solvent waste and increases laboratory throughput as compared to more conventional solvent extraction techniques. SPE clean-up followed the extraction step to remove high levels of chlorophyll and organic acids that may interfere with the resulting chromatographic analysis.[6] Sample extract clean-up was performed by cartridge SPE procedures utilizing a 500mg graphitized carbon black/500mg Primary Secondary Amine cartridge, to which MgSO4 was added to the top of the SPE cartridge at approximately half the height of the GCB/PSA bed. The cartridge was rinsed with 20mL of acetone. Then 0.5mL of the sample extract was added, 2.5uL of anthracene (recovery surrogate) was spiked onto the cartridge and carefully mixed by syringe. The solution was eluted with a 3:1 acetone:toluene mixture. The solution was then evaporated with nitrogen at 108°C until it was reduced to approximately 0.3mL. Toluene was added to adjust the final volume to 0.5mL. The samples were analyzed using various GC methods, which will be discussed in detail during the presentation, allowing for the potency analysis and the pesticide fingerprint to be determined in a single GCxGC separation.

References:

1. “DrugFacts: Marijuana.” Marijuana. National Institute of Drug on Abuse, Nov. 2010. Web. 30 July 2012. <http://www.drugabuse.gov/publications/drugfacts/marijuana>.
2. “Why Cannabis Testing Is Important to Patients.” Medical Marijuana Dispensary & Marijuana Doctors Directory. SC Laboratories, n.d. Web. 30 July 2012. <http://legalmarijuanadispensary.com/index.php?option=com_content>.
3. Bonsor, Kevin. “How Marijuana Works” 02 July 2001. HowStuffWorks.com.<http://science.howstuffworks.com/marijuana.htm> 30 July 2012.

 ** Presenting Author*

4. Stanford, Mahmoud A. ElSohly, Donald F., and Timothy P. Murphy. “Chemical Fingerprinting of Cannabis as a Means of Source Identification.” Marijuana and the Cannabinoids. Humana Press, 2007. Web. 30 July 2012. <http://www.hampapartiet.se/09.pdf>.
5. Cochran, Jack. “Screening for Bifenazate (Floramite) in Medical Marijuana Using QuEChERS and GC-FID Is It Possible? « ChromaBLOGraphy: Restek’s Chromatography Blog. RESTEK, 20 May 2012. Web. 30 July 2012. <http://blog.restek.com/?p=5068>.
6. Cochran, Jack, Julie Kowalski, Sharon Lupo, Michelle Misselwitz, and Amanda Rigdon. “High Quality Analysis of Pesticides in Marijuana Using QuEChERS, Cartridge SPE Cleanup, and GCxGC-TOFMS.” Restek Advantage. 2011.2 n. page. Web. 30 Jul. 2012. <http://www.restek.com/pdfs/GNAD1232-UNV_FLIPBOOK.pdf>.

Cannabinoids, GC/MS, GC-FID

 ** Presenting Author*