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## "Characterization and Abuse of Electronic Cigarettes: The Efficacy of "Personal Vaporizers" as an Illicit Drug Delivery System"

#### **FINAL REPORT**

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### **Statement of Problem**

Electronic cigarettes (e-cigarettes or e-cigs), known as "personal vaporizers" (PV) by avid users or electronic nicotine delivery systems (ENDS) by industry, have experienced a significant increase in popularity for those seeking an alternative to smoking traditional tobacco products. These products are comprised of a battery-powered atomizer and a cartridge filled with a pharmaceutical (nicotine), flavorings, and water dissolved in glycerol products. E-cigarette are manufactured in a variety of options: from off-the-shelf non-customizable devices to customizable, including self-wrapping of the element, homemade wicks, self-preparation of the e-cigarette liquid formulation (e-liquids), cups to hold plant material, dripping vs wicking, and wattage adjustors to administer the desired drug.

The lack of enforced regulation prior to May 2016 has resulted in easy accesses to e-cigarettes and has shepherded their nefarious uses. The use of the e-cigarettes as an illicit drug delivery device is touted on websites, forums, blogs, and videos describing how best to use them for specific illicit drugs such as tetrahydrocannabinol, methamphetamine, fentanyl, and heroin. These sites explain at length the benefits of "vaping" illicit drugs as it can conceivably be done in public without attracting notice. While some individuals and communities have begun to legislate where users can vape, vaping is not just acceptable, it is considered "cool" by many and often has the added benefit of no odor.

Analyzing paraphernalia for drug usage uses straightforward methodology established in controlled substance laboratories nationwide. E-cigarettes were largely uncharacterized at the beginning of this research. In forensic science laboratories, little is known or understood about their construction, let alone how they are used to deliver illicit drugs. From a general toxicological perspective, little is documented regarding the delivery of nicotine, particularly as a function of power, for-e-cigarettes. Even less is known regarding the adulteration of e-cigarettes and how the e-cigarettes are used or modified to optimize the delivery of an adulterant and/or alternative drug.

Problems can arise with using electronic cigarettes to deliver illicit drugs. According the vaping community, the dosing can be increased by turning up the wattage/voltage on the device. This method to increase dosage alone, or combined with increasing the volume of the "puff", could easily lead to overdoses. Increasing temperatures could lead to pyrolysis products which can potentially be used as biomarkers that could identify the use of e-cigarettes in biological tissues and which may have unknown biological effects. Drug forums are providing cautionary tales to users, however, these are overshadowed by the clear benefits these devices bring to drug users.

Few peer-reviewed published manuscripts in the literature describe, define, and/or illustrate the use of e-cigarettes. The overarching purpose for this study was to characterize the use and efficacy of electronic cigarettes to deliver pharmaceutical products. E-cigarettes were functionally described, methods were developed to analyze the pharmaceutical products (e-liquids and aerosol), truth in advertising was described for commercially available products, adulterations to the products were assessed, and potential biomarkers were evaluated.

### **Implications for Criminal Justice Policy and Practice**

#### What is the impact of the project on the criminal justice system?

Significant efforts have been made to educate law enforcement, medical examiners, toxicologists, and crime lab practitioners about drug use and abuse employing e-cigarettes. Public awareness has increased as to the potential dangers, and crime scene investigators and death investigators have

reported collecting these and sending them to crime labs. Ultimately, this study has provided greater understanding in the court systems nationwide as to the nature of drug usage, abuse, and overdose cases in which e-cigarettes were used to deliver an illicit drug.

We believe that the impact to the criminal justice system is slowly unfolding. However, the research group has been asked to speak at national and international conferences to continue to educate scientists and crime scene investigators as to the potential impact of using e-cigarettes illicitly. Dr. Peace, the PI, has been invited to numerous conferences to present our findings, and will be providing information directly to the DEA who now has significant interest in the adoption of e-cigarettes. Additionally, the research group has been interviewed for news outlets such as *New Scientist* and NPR's *With Good Reason*. This kind of broader exposure will benefit the criminal justice system from an immediate general awareness perspective to, hopefully, programmatic/systemic changes.

#### How has it contributed to crime laboratories?

Given that one role of the forensic toxicologist is to define and characterize drug usage trends, this publicly funded research posed an important, relevant, and critically timed study to address an identified threat to public health and criminal justice. This research supported the analytical efforts in controlled substances units and will support the findings and opinions of scientists, medical examiners, death investigators, and forensic toxicologists as they present analytical results.

As of April 24, 2017, the research group has been-consulted on seven e-cigarette cases. One case involved a professional athlete who submitted to a urine drug test and tested positive for a banned substance. He reported that he vaped from someone else's e-cigarettes, that contained an e-liquid purchased from a retail shop that was not labeled as having contained that substance. The laboratory who conducted the testing called to inquire about the feasibility of this claim. Based on our analyses of dozens of samples of e-liquids purchased from multiple vendors, the story was plausible. Three cases involved samples submitted through emergency room departments. One sample was determined to contain apomorphine and nuciferine, found in blue lotus flowers. Two cases involved potential overdose deaths. In the first, witnesses claimed that the victim vaped from an e-cigarette and died shortly thereafter. Analysis of the e-cigarette and e-liquid revealed no drug besides nicotine. One case involved a family-based litigation case in which someone claimed to be "just vaping" while illicit substances were found in the e-cigarette. And lastly, a "smoke easy" raid was conducted in a Philadelphia warehouse in which 22 people were arrested for marijuana possession and distribution. According to an analyst in the crime laboratory, the evidence collected included dozens of cartridges presumably containing cannabinoids. The analyst had attended the two webinars produced by the Center of Excellence on our research, and, as a result, was assigned the e-cigarette evidence. The research group will collaborate with that crime laboratory to analyze the products seized.

#### What is the impact on technology transfer?

In addition to providing critical information at an important time in the emergence of these products, the study has supported the efforts to understand the e-cigarette industry by regulatory agencies such as the FDA. Given that most of the knowledge of "puff topography", or the volume of cloud, duration of the inhale, and the concentration of drug in the cloud, is promulgated by users, this research has contributed to the scientific foundation for electronic e-cigarette usage.

Recently, the research group was asked to provide presentations and our list of publications to the U.S. Food and Drug Administration and to U.S. Department of the Navy, which is evaluating whether

or not to ban e-cigarettes on submarines. So, while the impact on the criminal justice system is evolving, significant implications exist in other forums.

## **Design Methods and Results**

The following aims and experiments were identified within the proposal. Within each, specific experimental goals and results are described.

In addition to pursuing these aims, we also monitor the current state of e-cigarette usage, messaging in the media, and legislation and regulation.

**Goal:** The goal was to identify information and resources to assist ongoing research to understand the forensic impact and implications of e-cigarettes. Information gleaned in the process of this study will assist decision-making regarding criminal justice and public education.

- Modifications and adulterations to electronic cigarettes and e-liquids, including illicit drug delivery, are described and promulgated by experienced users through videos, social media, and user blogs.
- All fifty states have developed policy in at least one of these five areas: sales to minors, defining e-cigarettes as tobacco products, bans on use in public, taxes, or other regulations.
- Media has reported deaths due to e-liquid consumption.
- Over the course of the 2+ year project, drug forum conversations evolved rapidly to discuss modifications and adulterations to e-cigarettes and e-liquids.
  - Recent conversations have included drugs such as Kratom (mitragynine), synthetic cannabinoids, synthetic opioids, and blue lotus (apomorphine and nuciferine)
  - Natural products in addition to illicit substances continue in the threads.
  - New e-cigarettes models emerged for the specific use of waxes, dabs, and dry natural products.
  - Modifications to the atomizers, mostly with regards to coil configurations, escalated as e-cigs became more technologically advanced.
  - Users continued to advocate regularly, despite prohibitions to public vaping, that the ease of transporting illicit substances and vaping illicit substances in public was a bonus for adopting the method.
- After the FDA banned the sale of products, websites had to stand-up a screening process to make sure that people were >18 years old. We monitored the complexity of this and have been challenged by more reputable websites who have questioned the identity of the purchaser. Since the credit card had the university address, the vendor was unable to "confirm" identity and age of the purchaser. Vendors with questionable e-liquid products have had no problem with the disconnect between the address of the credit card and our identity. While there is no way to know without further investigation, the difference between the two types of vendors has been observed.
- An evaluation of historical patents was performed to create a timeline of e-cigarettes and their mechanisms for operation, particularly as their construction evolves.

### Aim 1

Develop reliable, validated analytical methods to support the efficient and thorough analysis of e-cigarette devices, device components, and aerosol for pharmaceuticals in adulterated, unadulterated, and self-prepared formulations. *Experiment 1.* Develop method for capturing vapor product from electronic cigarettes by water trap and solid-phase micro-extraction (SPME).

<u>Experiment 2.</u> Develop validated method for the qualitative assessment of electronic cigarettes artifacts using Direct Analysis in Real Time Accu-TOF<sup>TM</sup> Mass Spectrometry (DART). A quantitative method will also be assessed.

Goal: To develop a simple trap to analyze the aerosol from e-cigarettes.

**Results:** A simple trap was developed and validated to analyze aerosol via SPME and to trap ingredients/drugs in water. Erlenmeyer flasks were connected to a vacuum with tubing and glass wool filters in between for capture. Corks were drilled to accommodate the tubing attached to the e-cigarette and a septum for the SPME sampling (Figure 1). Briefly, two Erlenmeyer flasks were connected in tandem to a vacuum with a flow rate of 2.3 L/min. DI-water was added to each trap and a gas dispersion tube bubbled the aerosol into the water. Glass wool was placed in between the two traps to contain the aerosol in the first trap. Results are reported in Aim 2.

**Goal:** To develop a sampling protocol in order to use solid-phase microextraction (SPME) to capture aerosol produced by an e-cigarette for analysis by a SPME GC-MS and SPME-DART-MS methods in order to both qualitatively and quantitatively assess the amount of nicotine and flavoring agents found in e-cigarette aerosol.

**Results:** Polydiethylsiloxane (PDMS) SPME fibers were demonstrated to successfully capture nicotine and flavoring agents in concentrations that were analyzed by GC-MS and DART-MS. The aerosol produced by the e-cigarette was trapped with the apparatus described above. A 7 $\mu$ m or 10  $\mu$ m PDMS SPME fiber was inserted through a stopper in the first trap, depending on the molecular weight of the drug. The fiber was introduced into the trap while the e-cigarette was activated for four seconds, aerosolizing 7-10  $\mu$ L and the aerosol filled the trap. The SPME fiber was held in the trap for five minutes, after which the fiber was removed.

The SPME fiber was then inserted into the injection port of an Agilent GC-MS 6890N/5973 Mass Selective Detector with an HP-5MS column 30 m  $\times$  0.25 mm id  $\times$  0.25 µm. The injection port was set to 315 °C and the run was made in splitless mode with a 15-minute thermal desorption time. The initial temperature was set to 120 °C, with a ramp to 300 °C at 10 °C/min, with a hold time of 12 min at 300 °C, for a total run time of 30 min. The fibers were thermally cleaned between runs to ensure no carryover occurred between samples. (Figure 2).

Alternatively, the SPME fiber was waved in the helium stream of the DART-MS following previously validated methods. The analysis was performed in positive-ion mode with a helium stream temperature of 300 °C. The ion guide peak voltage was 400 V, reflectron voltage was 900 V, orifice 2 was set to 5 V, and the ring lens was set to 3 V with orifice 1 operated in function switching mode at 20, 60, or 90 V with a single data file created for all three voltages. The range of masses measured was from 40 to 1100 Da. (Figure 3).

The general analytical scheme adopted in the laboratory was that all e-liquids were screened by DART-MS. Samples were then quantitated by GC-MS or LC-MS/MS. Samples were aerosolized and active ingredients were extracted by SPME for analysis by DART-MS and GC-MS in order to demonstrate that pharmacologically active ingredients were in the vapor. See Figure 4 for examples.

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#### *Experiment 3. Quantify nicotine and adulterant pharmaceuticals by LCMS<sup>3</sup>*

Goal: Validated method for nicotine and Drugs Other Than Nicotine (DOTNs) by LCMS<sup>3</sup>

**Results:** The nicotine concentrations of the e-liquids were determined using HPLC-MS/MS with a Hypersil® Gold 3x50 mm,  $5 \mu \text{m}$  column and a method previously published. The mobile phase consisted of 1:9 DI water with 10 mmol ammonium acetate and 0.1% formic acid: methanol, and was run at an isocratic flow rate of 0.5 mL/min. The injection volume was 10  $\mu$ L. The ion spray voltage was 5000 V and the source temperature was 600 °C with 30 mL/min curtain gas flow. The instrument was operated in multiple reaction monitoring mode (MRM) monitoring the following m/z transitions: nicotine, 163>130 and 163>117; and nicotine-d4, 167>134. A linear regression was plotted of peak area ratio of nicotine to internal standard versus nicotine concentration.

A method for quantitation of methamphetamine was validated using an Agilent 6890N Gas Chromatograph coupled to a 5973 Mass Selective Detector per SWGTOX guidelines. Chromatographic separation was performed on a HP-5MS column 30 m x 0.25 mm id x 0.25 mm (Agilent, Santa Clara, CA). The GC/MS was operated in a split mode at a ratio of 6:1, with 1 µL injection volume. The carrier gas was helium at a linear velocity of 35 cm/s. The GC had an oven temperature of 120 °C-200 °C at a rate of 10 °C/min then ramped to 280 °C at a rate of 30 °C/min. The MSD was operated in SIM mode with 58, 64, 91, 96, 134 m/z as the selected ions. Quantification was performed using 58 and 64 m/z as the quantitative ions for methamphetamine and methamphetamine-d11, respectively. The qualitative ions for methamphetamine were 91 and 64 m/z and 91 m/z for methamphetamine-d11. The total run time of 10.67 minutes. A six-point calibration curve was constructed with methamphetamine concentrations of 100, 200, 500, 750, 1000, and 2000 ng/mL with 500 ng/mL of methamphetamine-d11 as internal standard. The calibration curve was matrix-matched and was extracted from water using the method previously described. A linear regression was generated using the peak area ratio counts of methamphetamine and internal standard versus the theoretical calibrator concentrations, and  $r^2 > r^2$ 0.9981 for all curves. The limit of quantitation was administratively set to 100 ng/mL. Six sets of controls were included with each analytical batch: Limit of Detection QC (100 ng/mL), Low QC (150 ng/mL), Mid QC (600 ng/mL), High QC (1500 ng/mL), a blank, and a double blank. Intraday precision and bias were determined by the largest percent coefficient of variation (% CV) and by the largest percent difference of the five runs (N=5). Carryover was assessed by injecting the lowest quality control (150 ng/mL) following the injection of the high-quality control (1500 ng/mL). (Figure 5).

A method for the quantitation of methadone was validated using an Agilent 6890N Gas Chromatograph coupled to a 5973 Mass Selective Detector per SWGTOX guidelines. Chromatographic separation was performed on a HP-5MS column 30 m x 0.25 mm id x 0.25 mm (Agilent, Santa Clara, CA). The GC/MS was operated in a split mode at a ratio of 20:1, with 1 µL injection volume. The carrier gas was helium at a linear velocity of 20 mL/min. The GC had a front inlet temperature of 275 °C. The temperature program began with the oven temperature of 225 °C, and then programmed at a rate of 15 °C/min to 285 °C. The MSD was operated in SIM mode with 72, 223, 294, and 309 m/z as the selected ions for methadone and 78, 226, 303, and 318 m/z for methadone-d9. A seven-point calibration curve ranging from 100-5000 ng/mL of methadone was prepared along with a blank, a double blank control, and controls containing methadone and its internal standard were analyzed. The controls were run in triplicate with Limit of Detection/Quantitation control at 100 ng/mL; a low-control at 150 ng/mL; a mid-control at 1000 ng/mL; and a high-control at 4500 ng/mL. The limit of detection was arbitrarily set to match the limit of quantitation at 100 ng/mL. An internal standard (500 ng/mL methadone-d9) was added to each calibrator, blank, controls, and sample. The coefficient of determination  $(r^2)$  was  $\Box$  0.9985 for all calibration curves. The bias for all the controls (n=3) for methadone ranged from 5-15% with coefficient of variance (% CV) of 20%. Carryover was assessed by injecting the lowest quality control following the injection of the high-quality control.

*Experiment 4. Develop a dynamic headspace GCMS (DHS GCMS) method for the direct analysis of e-cigarette components.* 

**Challenge:** The DHS GC-MS had significant equipment failures over many months. A method for headspace analysis used in the analysis of fire debris evidence was concept tested to analyze atomizers on e-cigarettes. It was demonstrated that this method could be adopted for nicotine analysis. However, this method did not work for the analysis of methamphetamine. Static headspace with the charcoal strip used in nicotine analysis was evaluated a number of times with a number of extraction time periods and temperature parameters for methamphetamine analysis. Methamphetamine was demonstrated to aerosolize; however, it was not extracted from the aerosol onto the charcoal test strip or it was not possible to extract it from the charcoal test strip. The conclusion is that the charcoal test strip was not suitable for some drugs.

#### Aim 2

# Characterize representative electronic cigarettes available for consumers to purchase, to include efficacy of drug delivery.

<u>Experiment 1.</u> Characterize major classes of e-cigarette devices available for consumers to purchase and describe the function of each component.

**Goal:** The three major types of e-cigarettes were characterized (Figure 6-9). In order to understand the relationship between voltage, resistance, heat, and concentration of drugs in the aerosol, coil temperatures were assessed at various voltages and various gauges.

**Method:** Two popular types of wire used within the e-cigarette community are Kanthal A-1 and Nichrome 80:20. Coils can be either contact or non-contact. Contact coils are tightly wrapped and the wire touches each wrap. Non-contact coils do not touch and typically have the wraps evenly spaced apart from one another. The temperature of the coils for the dry burns and wet burns when the coil is wrapped to the same resistance and burned at the same voltages were assessed. Both metal types will be evaluated at the same resistance (1.8  $\Omega$ ), wet and dry, contact and non-contact, with wire gauge ranging from 26 to 36 and at 3.5, 3.7, 4.2, 4.5, 5.0, 5.5, and 6.0 volts, with replicates of at least 3 for each variable. The atomizer was a Kayfun Lite Close Atomizer. The type of atomizer is less important than the assessment of the coils as the atomizer was attached to a constant power supply instead of a battery. Center coil temperatures of two wire types, each in three gauges, wrapped in both contact and non-contact, fired at four voltages, and wet with 100% VG or kept dry were measured. All coils were hand-built to 1.8  $\Omega$  resistance. Temperatures were measured using a Micro-Epsilon IR sensor with a laser sighting.

To complicate this single coil model, users have begun to use dual coils in parallel for a technique called "sub-ohming" (<1  $\Omega$ ). As opposed to a single coil being wrapped to a resistance of 1.8  $\Omega$ , 2 coils are each wrapped to about 1.2  $\Omega$  and are aligned in parallel to create a resistance of 0.6  $\Omega$ 

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(Figure 10). In order to explore if there was potentially a significant difference between the 2 types of atomizers, a short study was conducted.

**Results:** In the dry configuration, Kanthal is the hotter wire, meaning it is able to sustain hotter temperatures. This is not surprising since it is an alloy composed of iron, chromium, and aluminum and has a melting point of 1400°C. Nichrome is a nickel and chromium allow with a melting point of 1200°C. Not surprisingly, contact is hotter than non-contact.

When coils had a wick wet with 100% vegetable glycerin (VG), no statistically significant difference was seen between wire gauge, wire type, and wire configuration. Hence, VG is a limiting factor to coil temperature. (Figures 11-12)

These findings are significant because they provide some basis for understanding the vaporization of drugs into the aerosols. More importantly, the temperature of the wet coils disproves user claims that the wet coils get hotter with Kanthal. The real value of using Kanthal is that the melting temperature is higher for those who are vaping off dry coils. While temperature may be the same, it is unknown as to if the wire type and size may be impacting the particle size of the aerosols.

When the coils are in contact with 100% propylene glycol (PG), the temperature output between the two mediums differed significantly. The PG medium had lower temperature outputs than VG, reaching temperatures as low as 160 °C and as high as 360 °C. The type of coil, Kanthal A-1 or nichrome 80:20 did not have a significant difference in temperature nor did differing coil configurations. There was a trend of temperature increase with decreasing wire thickness. These results contradict what most e-cigarettes users claim: That wire type and configuration have an impact on the temperature outputs of the coils and how they vaporize the e-liquid. It appears that the medium that the coils are submerged in have a more significant impact in the temperature outputs of the coils, which may also lead to an impact in the particle size produced.

The temperature profile of the dual dry 26 gauge Kanthal coils wrapped in contact from 3.5 to 5.0 V showed minor temperature variations between the two coils. This is predominantly due to the nature of how much wire is touching the contacts. Microns of metal length can impact resistance enough to change the temperature profile between the two coils in the same atomizer. The temperature profile for the 26 gauge Kanthal dual coil sub-Ohm at 3.7 V is significantly higher than what was found with the 26 gauge Kanthal single coil 1.8  $\Omega$  at 3.7 V wire (951 °C and 793 °C, respectively), both wrapped in contact and dry, due to the difference between resistances of the coils. By holding the voltage constant between the two e-cigarette models, the current running through the lower resistance dual coil model is higher, leading to higher wattage and higher temperatures.

<u>Experiment 2.</u> Characterize the delivery of nicotine in e-cigarettes, based upon device type and voltage, in terms of concentration of nicotine in the liquid and concentration of nicotine in the vapor. After this is characterized, adulterant illicit drugs will also be evaluated (THC, methamphetamine, opiates, and other drugs as necessary and time permits)

**Goal:** Characterize the concentration of nicotine in aerosol based on the performance of the device, including varying the voltage to manipulate the heat produced by the heating element.

**Results:** Studies showed that the yield of nicotine in the aerosolized e-liquid consisting of 50:50 propylene glycol:vegetable glycerin (PG:VG) with 12 mg/L nicotine (delivered by a KangerTech

AeroTank, 1.8  $\Omega$  preassembled atomizer with eGo-V2 variable voltage battery e-cigarette, Nichrome, 34 gauge, non-contact coil) increased with increasing voltage (3.9, 4.3, and 4.7 V), from 89 µg to 125 µg (based on weight difference of the tank) (see figure). The concentration of nicotine recovered from the traps, as determined by LC-MS<sup>3</sup>, averaged 378 ng/ml (+/- 64). As seen with the nicotine yield, the aerosol concentration increased from 322 to 374 to 432ng/ml, at 3.9, 4.3, and 4.7 V, but had moderately high SD. The average recovery of nicotine in the trap across the voltages was 101%, ranging from 82-113%. (Figure 13)

See Aim 1 for analysis of methamphetamine in e-liquids. A study was conducted to determine if increasing voltage of the e-cigarette yielded higher dosing in the aerosol. The study concluded that there was no significant difference in the concentration of methamphetamine as the voltage of the e-cigarette increased, serving to contradict the users' myth. Three concentrations of methamphetamine were prepared in 50:50 PG:VG (v:v) and aerosolized at 3.9, 4.3, and 4.7 V for 4 seconds at 2.3 ml/minute. Five replicates were analyzed at each voltage. The change in voltage did not significantly alter the dose of methamphetamine contained in the aerosol. (Figure 14)

For methadone, the theoretical dose capture concentrations were determined from the gravimetric difference of the e-cigarette tank before and after aerosolization into the trap. At 10 mg/mL of methadone in the e-liquid, the dose per puff was expected to from 38 to 46  $\mu$ g with increasing voltage. For the 30 mg/mL e-liquid, the predicted concentrations ranged from 170 to 221  $\mu$ g and the 60 mg/mL e-liquid from 234 to 549  $\mu$ g. GC/MS results showed that at 10 mg/mL, the average dose per puff at 3.9 and 4.7 V was 8 and 4  $\mu$ g respectively with a 19 and 8% recovery. At 30 mg/mL the average doses were 37 and 17  $\mu$ g with 16 and 10% recovery, and at 60 mg/mL, the average doses were 30 and 417  $\mu$ g with 12 and 79% recovery. Statistical analysis of ANOVAs comparing the dose capture results by voltage showed that there was not a difference between the dosage at 3.9 and 4.3 V (P= 0.76), but that there was a difference in the dosage between 3.9 and 4.7 V and 4.3 and 4.7 V (P <0.001). The hypothesis regarding the low recovery of methadone from the aerosol is that the boiling point for methadone is higher than the temperature of the coil in the electronic cigarette.

# *Experiment 3.* Characterize the formation of potential biomarkers formed during pyrolysis based on the wattage of the e-cigarette.

**Goal:** Characterization of coil temperature studies have revealed that the coils, after multiple burns begin to deteriorate. A study to characterize the degradation of the coils by SEM-EDX was conducted.

**Results:** Nichrome and Kanthal wires at 30, 32, and 34 gauges were triggered 1, 50, and 150 times in both contact, and non-contact coil configurations. The voltage used was dependent on the gauge and coil configuration and can be found in table 1. All coils were triggered dry for 10 seconds, and SEX-EDX analysis was completed at 2000x magnification. Two metals for each wire type showed a significant decrease at 1, 50, and 150 burns as compared to a pristine (unburned) coil. Kanthal wires showed a significant decrease in iron and nickel. Statistical analysis shows that there is no significant difference in iron or nickel loss between gauges for nichrome wires, while Kanthal wires were found to have significantly less loss of iron and chromium with 30 gauge wire as compared to 32 and 34 gauge wire. The data also suggests that Kanthal coils wrapped in a contact configuration. Nichrome wires, however, show a general trend of no difference in nickel and iron loss when comparing final metal percentage of contact and non-contact

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configurations. Overall, the data has shown that the metal composition of both nichrome and Kanthal wires decreases significantly after 1 burn and progressively deteriorates as the number of burns increases. This effect is independent of wire type, gauge, and configuration. (Figures 15-17)

A method is being validated on the Agilent 7700 ICP-Mass Spectrometer to analyze metals in the aerosol. A filter system had to be developed to capture the aerosol. Using the e-cigarette system as previously described, ten 30 gauge Kanthal and Nichrome wires were heated for 10 seconds and the aerosol was each trapped onto a single 0.1 micron Teflon hydrophobic filter. This was repeated 10 times. They were digested in aqua regia, diluted, and analyzed by ICP-MS. Samples were run to eliminate the potential that metal would be in the aerosol and does not condense in the mouthpiece of the atomizer. Average ppb of chromium-53, iron-57, and nickel-60 for Kanthal was  $11.0 (\pm 15.4), 41.9 (\pm 58.7), and 4.04 (\pm 5.60), respectively.$ 

#### Aim 3

# Characterize the liquid refill products for electronic cigarettes, to include nicotine and adulterant pharmaceuticals.

#### *Experiment 1.* Characterize variation in supplies purchased by consumers:

**Goal:** Characterize the e-liquids for e-cigarettes, to include nicotine and adulterant pharmaceuticals (THC, methamphetamine, heroin).

**Results:** A stability study was conducted to understand the quality of e-liquids and how they might impact nicotine concentration. Samples were stored in three types of containers in three conditions: 24 hours light at room temperature (RT), 24 hours dark at room temperature, and 24 hours dark at 40 °C. They were samples periodically and analyzed by LC-MS/MS. The concentration of nicotine decreased significantly from time 0 through time 12 weeks, and then plateaued for the remaining time when exposed to light conditions. For example, the 36 mg/mL sample kept in 24 hour light at room temperature decreased to 27.8 mg/mL in 12 weeks. The container type (glass, plastic, and amber glass) did not impact the concentration of nicotine in the sample. The concentration of nicotine in commercial samples deteriorated from 20.5 mg/mL to 13.2 mg/mL in 12 weeks and then plateaued. However, no statistically significant difference exists between light and RT, dark and RT, and dark and refrigerated (Figure 18).

Goal: Characterize e-liquids purchased online for "truth in advertising"

**Results:** Nicotine concentrations of 27 commercial e-liquids were compared to advertised nicotine concentration (Table 1). Actual concentrations varied 53-139% from advertised. Glycol ratios in the 27 commercial e-liquids were compared to advertised label concentration and found to be in general agreement.

A sample of e-liquid was received in the laboratory labeled as a cannabinoid-based products. The concentrations of the THC, 42.6% (v:v), and CBD, 0.5% (v:v), were different then the labeled content, THC at 69% and CBD at 1%. Four additional unlabeled cannabinoids were identified and quantitated. Fourteen terpene compounds were identified:  $\alpha$ -pinene,  $\beta$ -myrcene,  $\beta$ -pinene, limonene, (1R)-endo-(+)-fenchyl alcohol, linalool,  $\alpha$ -terpineol,  $\beta$ -caryophyllene,  $\alpha$ -humulene, cis/trans nerolidol, guaiol, (+)-cedrol, and  $\alpha$ -bisabolol. The identification of these unlabeled cannabinoids and marijuana terpenes indicated the e-liquid was produced by extraction of marijuana.

Cannabidiol (CBD) was listed as the active ingredient in e-liquids found online. Two e-liquids were acquired that claim to be infused with CBD and contained 3.3 mg/mL CBD. They screened positive by DART-MS and CBD was quantitated by LC-MS<sup>3</sup> at 6.53 and 7.61 mg/mL. The website (cloud9hemp.com) has an FDA Disclosure statement that reads "...FDA considers non-THC based hemp products to be "food based" and therefore legal...CBD Rich Hemp Oil is legal in all 50 states." They also state that the products are "not intended to diagnose, treat, or cure any disease."

Ethanol was determined in 53 of the 56 e-liquids by HS GC-FID. Only one e-liquid included ethanol as a labeled component. Thirty of 42 e-liquid samples were reported as positive for ethanol with concentrations > 3.70 g/mL. Once it was determined that commercial e-liquids contain ethanol, coil temperatures were evaluated for any significant change from the original temperature studies conducted without ethanol in lab-prepared e-liquids. Temperatures were significantly lower in some cases and higher in others. Therefore, it could be said that the temperature the coils were unpredictable with ethanol in the e-liquid. (Figure 19)

## **Project Findings**

#### <u>Aim 1</u>

Develop reliable, validated analytical methods to support the efficient and thorough analysis of e-cigarette devices, device components, and aerosol for pharmaceuticals in adulterated, unadulterated, and self-prepared formulations.

- 1. DART-MS, LC-MS<sup>3</sup>, and GC-MS methods were successfully validated to screen, confirm, and quantitate pharmacologically active ingredients in e-liquids and the aerosol.
  - a. A trap and SPME extraction method were developed and validated to successfully capture the aerosol produced by an e-cigarette.

#### <u>Aim 2</u>

Characterize representative electronic cigarettes available for consumers to purchase, to include efficacy of drug delivery.

- 2. Increases in voltage do not increase the temperature of the coil significantly and do not yield significantly higher concentrations of the pharmacologically active ingredient (nicotine or methamphetamine) in the aerosol.
- 3. Metal composition of both nichrome and Kanthal wires decreases significantly after 1 burn and progressively deteriorates as the number of burns increases. This effect is independent of wire type, gauge, and configuration.

### <u>Aim 3</u>

Characterize the liquid refill products for electronic cigarettes, to include nicotine and adulterant pharmaceuticals.

- 4. Given that the e-cigarette industry is un-regulated, it should not be unexpected to find nicotine or glycol concentrations different than the labeled concentrations on e-cigarette liquid formulations.
- 5. Poor QA/QC in facilities producing e-liquid products result in products that have significantly less or significantly more than what is indicated on the bottle.
- 6. Some e-liquids are not labeled as to the pharmacologically active component, nor are they advertised by the vendor. Indicators that a product contains a DOTN is that it is 2-10 times more expensive than a nicotine-based e-liquid.
- 7. Ethanol was a major component in most e-liquids and was only labeled in one product.

## **Future Studies**

- 1. It is imperative to evaluate how vaping alcohol impacts field sobriety tests and BAC.
- 2. It is important to continue to monitor how the technology for e-cigs continues to evolve. In the 2.5 years of this study, the types of e-cigarettes have grown significantly in order to vape dry herb, waxes, dabs, oils, and other types of tinctures.
- 3. Eutectic mixtures should be explored to assess improving the aerosolization of high molecule weight molecules, such as opioids, particularly since user blogs indicate recommendations for cutting these drugs with caffeine before putting into e-liquids.

## **Dissemination/Products/Accomplishments**

This research group has aggressively presented and published its finding. Twenty scientific presentations have been made (Table 2). Two traditional workshops and two webinars were conducted. Six peer-reviewed manuscripts have been published, and three more are in preparation and on-track for submission by June.

Findings from this study were also disseminated in China, but NIJ provided no funding for this travel.

#### Presentations – Workshops and Webinars

<u>Peace MR.</u> "Characterization and Abuse of Electronic Cigarettes: The Efficacy of Personal Vaporizers as an Illicit Drug Delivery System." Forensic Technology Center of Excellence Live Webinar, August 2016.

<u>Peace MR</u>, Butler KE, Stone JW. "Characterization and Abuse of Electronic Cigarettes: The Efficacy of Personal Vaporizers as an Illicit Drug Delivery System." NIJ Research Symposium, February 2016.

"Vaping: What you don't know about electronic cigarettes and why you should care"

- Society of Forensic Toxicologists Annual Meeting, Dallas, TX, 2016, Chair: <u>Michelle Peace</u>, Co-Chair: Justin Poklis
- American Academy of Forensic Scientists, Las Vegas, NV, 2016, Chair: <u>Michelle Peace</u>, Co-Chair: Justin Poklis

#### Invited Talks, Media Engagements, Miscellaneous

- Eastern Analytical Symposium. "New Marijuana Products" in a workshop titled "Marijuana: From Research and Use to Abuse", Princeton, NJ, November 2017.
- Peace MR, Poklis JL. "Chasing the E-Cigarette Dragon: Overview of Research Findings". DEA, July 2017
- <u>Peace MR.</u> "Current Trends in Drug Addiction and Trafficking in the United States" and "Electronic Cigarettes and the Threat to Criminal Justice and Public Heath" International Conference on Forensic Science, University of Public Safety, Beijing, China, and Northwest Law University, Xi'An, China, September 2016 (NOT FUNDED BY NIJ)
- <u>Peace MR.</u> "Characterization and Abuse of Electronic Cigarettes: The Efficacy of Personal Vaporizers as an Illicit Drug Delivery System." Tobacco Product Analysis Technology Summit, Research Triangle Park, Raleigh, NC, July 2016.
- Interview with <u>New Scientist Magazine</u> published December 14, 2016
- Interview with National Public Radio "With Good Reason" in December, to air in January

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#### **Manuscripts**

- Poklis JL, Wolf, CE, <u>Peace MR</u>. Ethanol Concentration in 56 Refillable Electronic Cigarettes Liquid Formulations Determined by Headspace Gas Chromatography with Flame Ionization Detector (HS-GC-FID). Drug Testing and Analysis DOI: 10.1002/dta.2193
- Poklis JL, Mulder HA, Halquist MS, Wolf CE, Poklis A, <u>Peace MR.</u> The Blue Lotus Flower (Nymphea Caerulea) Resin Used in a New Type of Electronic Cigarette, the Re-buildable Dripping Atomizer. Journal of Psychotropic Drugs (2017). DOI: 10.1080/02791072.2017.1290304
- <u>Peace MR</u>, Krakowiak RI, Wolf CE, Poklis A, Poklis JL. Identification of MDMB-FUBINACA in Commercially Available E-liquid Formulations for Use in Electronic Cigarettes. Forensic Science International (2016), doi.org/10.1016/j.forsciint.2016.12.031
- Peace MR, Butler KE, Wolf CE, Poklis JL, Poklis A. Evaluation of Two Commercially Available Cannabidiol Formulations for use in Electronic Cigarettes, Frontiers in Pharmacology, August 29, 2016. doi: 10.3389/fphar.2016.00279.
- 5. <u>Peace MR</u>, Stone J, Poklis JL, Turner JBM, Poklis A. Analysis of a Commercial Marijuana E-cigarette Formulation. Journal of Analytical Toxicology, 40(3); April 2016. doi: 10.1093/jat/bkw021
- Peace MR, Baird T, Poklis JL, Wolf CE, Smith N, Turner JBM, Poklis A. Concentration of Nicotine and Glycols in 27 E-Cigarette Formulations. Journal of Analytical Toxicology, 40(6); May 2016. doi: 10.1093/jat/bkw037
- 7. <u>Peace MR</u>, Baird TR, Stone JW, Butler KE, Poklis JL, Poklis A. Capture and Quantification of Nicotine Generated by an Electronic Cigarette by SPME-GC-MS and HPLC-MS/MS. In preparation.
- 8. <u>Peace MR</u>, Stewart JB, Mulder HA, Dupont A, Royals J, Forsythe K, Poklis JL, Turner JBM. Assessment of Temperature and Metals Loss from Electronic Cigarette Coils with Changing Voltages. In preparation.
- 9. Peace MR, Mulder HA, Poklis JL. The History and Evolution of Electronic Cigarettes. In preparation.

## Appendix

## **Figures and Tables**

Figure 1. Diagram of trap to capture aerosol from electronic cigarette.



**Figure 2.** Gas Chromatograph of commercial e-liquid VapeWell Cheery. Major components of the vapor detected were benzaldehyde, d-limonene, and nicotine.



**Figure 3.** Mass Spectrum of SPME-DART analysis of VapeWell Cherry. The components detected were propylene glycol, benzaldehyde, limonene, and nicotine.



**Figure 4.** SPME GC-MS results for aerosolized (a) CBD, (b) methamphetamine and nicotine, and (c) MDMB-Fubinaca.



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#### Figure 6. Cig-a-like electronic cigarette.

Electronic cigarette is not customizable and have a self-contained non-modifiable e-liquid.



#### Figure 7. Clearomizer electronic cigarette components.

I. (a) Battery power supply (b) Clearomizer tank (c) Mouthpiece (d) Atomizer base. II. (a) Heating element (b) Wick.



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#### Figure 8. Cartomizer tank electronic cigarette

I. (a) Variable voltage power supply (b) cartomizer tank (c) Steel cartomizer (d) Mouthpiece. II (a) Steel cartomizer, (b) Wicking material, (c) heating element



#### Figure 9. Rebuildable atomizer electronic cigarette.

I. (a) Variable voltage power supply (b) Mouthpiece, (c) Base containing heating element and wick, (d) Tank. II. (a) wicking material (b) heating element.



**Figure 10.** Single coil atomizer (on left) versus the Rebuildable Dripper Atomizer (RDA) with 2 coils wrapped in parallel for "sub-ohming" technique.



Figure 11. Temperature of center coil of 3 gauges of wire that were dry (top) or wet with 100% VG (bottom)



Figure 12. Comparison of temperature output of Nichrome wires in 100% VG (left) and 100% PG (right)



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**Figure 13.** Nicotine yield as a function of voltage of the KangerTech AeroTank,  $1.8 \Omega$ 

Figure 14: Quantitation of methamphetamine at three voltages.



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#### Figure 15. Percent weight of metals lost following 0, 1, 50, and 150 burns

**Figure 16.** Kanthal contact 30 AWG surface features at 2000x magnification on the SEM following 0, 1, 50, and 150 burns







New

1 Burn

50 Burns

150 Burns

**Figure 18.** Concentration of nicotine in eliquids stored at 3 conditions: Light + Room Temperature (LR), Dark + Room Temperature (DR), and Dark + Cold (DC).



Brand / Flavor	Label (mg/mL)	Mean (mg/mL)	S.D. (mg/mL)	%CV	Accuracy
258 Rally Squirrel	16	10.3	0.1	1	64
Captain Ron	12	11	0.2	2	92
Cheery	18	14.7	0.9	6	82
Delta	12	7.7	1.1	14	64
El Kamino	12	8.7	0.7	8	73
FennetHIGH	12	12.4	0.2	2	103
Grandmaster	6	7.8	0.4	5	131
Gremlin Juice Birthday Cake	12	13.8	3.1	23	115
Grumpy's Hooch	12	10	1.9	19	84
GWAR Spew	12	14.6	2.4	16	122
Indigo Birthday Cake	12	10.8	1.1	10	90
Jango	12	12.6	1.2	10	105
Kentucky Mint Julip	6	6.3	0.7	11	105
Mayflower	6	4.9	1.1	22	82
Peach Tobacco	12	8.9	0.7	8	74
Pharaoh	12	10.7	0.9	9	89
Snake Eyes	12	10.1	1.1	11	84
Snake Oil	12	10.5	1.5	14	87
Spearmint	22	11.6	2.7	23	53
Sunset	6	6	0.5	9	100
Turkish Tobacco	12	11.2	1.2	11	93
Unflavored PG	6	8.3	1.3	16	139
Vanilla Cream Custard	6	6	1.2	20	99
Vanilla Custard	12	10.7	2.7	25	89
Vanilla Tobacco	6	7.8	2.3	30	130
VG (12 mg/mL)	12	10.3	0.2	2	85
White Gummy Bear	6	5	0.4	8	83

## Table 1: Nicotine Quantitation by HPLC-MS<sup>3</sup>







#### **Table 2 Scientific Presentations Over Life of Funding**

- Royals JM (presenter), Poklis JL, Turner JBM, Wolf CE, <u>Peace MR</u>. Ethanol in E-liquids: Concentration in 35 Formulations by Headspace Gas Chromatography with Flame Ionization Detector (HS-GC-FID) and the Impact on the Temperature of the E-Cigarette Coils. Poster Presentation, Society of Forensic Toxicologists, Boca Raton, FL, 2017.
- Krakowiak R, Poklis JL, Turner JBM, Poklis A, Davis LS, Mulder HA (presenter), <u>Peace MR</u>. Quantitation of Aerosolized Methamphetamine from Electronic Cigarettes GC/MS: Does increasing the voltage increase the dose? Poster Presentation, Society of Forensic Toxicologists, Boca Raton, FL, 2017.
- Joiner RK, Bohidar N, Kirby BF, <u>Peace MR</u>, Ward BC (presenter). Rapid field testing of nicotine in e-liquids. Platform Presentation, Society of Forensic Toxicologists, Boca Raton, FL, 2017.
- Peace MR (presenter), Mulder HA, Krakowiak R, Turner JBM, Halquist MA, Wolf CE, Poklis JL, Poklis A. An Assessment of Drugs Other Than Nicotine (DOTNs) in Electronic Cigarette Products. Poster Presentation, PittCon, Chicago IL, 2017.
- Poklis JL (presenter), Wolf CE, <u>Peace MR</u>. Ethanol Concentration in 63 Refillable Electronic Cigarettes Liquid Formulations Determined by Headspace Gas Chromatography with Flame Ionization Detector (HS-GC-FID). Platform Presentation, PittCon, Chicago IL, 2017.
- 6. Stewart J (presenter), Turner JBM, Poklis JL, Poklis A, <u>Peace MR</u>. Metal Composition of Electronic Cigarette Coils Pre- and Post-Heating by Scanning Electron Microscopy. Poster Presentation, PittCon, Chicago IL, 2017.
- 7. Patterson JL (presenter), Poklis JL, Hindle M, Turner JBM, Wolf CE, Poklis A, <u>Peace MR</u>. Evaluation of the Nicotine Particle Size in an Aerosol Formed by an Electronic Cigarette. Platform Presentation, PittCon, Chicago IL, 2017.
- Krakowiak RI (presenter), Poklis JL, Wolf CE, Poklis A, <u>Peace MR</u>. An Analysis of E-liquids Containing MDMB-Fubinaca. American Academy of Forensic Sciences. New Orleans, LA, 2017.
- 9. Patterson JL (presenter), Poklis JL, Hindle M, Wolf CE, McGee Turner JB, Poklis A, <u>Peace MR</u>. Evaluation of the Particle Size in the Aerosol Produced by an Electronic Cigarette. Platform Presentation Society of Forensic Toxicologists, Dallas TX, 2016
- Krakowiak RI (presenter), Butler KE, Stone JW, Baird TR, Poklis JL, Turner JBM, Poklis A, <u>Peace MR</u>. Analysis of Aerosolized Methamphetamine Infused E-Liquids by Solid Phase Microextraction using Gas Chromatography Mass Spectrometry (SPME-GC-MS), Direct Analysis in Real Time AccuTOFTM Mass Spectrometry (SPME-DART-MS), and Headspace Gas Chromatography Mass Spectrometry (HS-GC-MS). Poster Presentation Society of Forensic Toxicologists, Dallas TX, 2016
- Mulder HA (presenter), Blue IP, Poklis JL, Patterson JL, Krakowiak RI, Forsythe K, Royals J, Dupont A, Poklis A, <u>Peace MR</u>, Turner JBM. Temperature Characterization of Electronic Cigarette Atomizer By Infrared Temperature Sensing. Poster Presentation Society of Forensic Toxicologists, Dallas TX, 2016
- 12. Blue IP (presenter), Butler KE, McGee Turner JB, <u>Peace MR</u>. That's Not Fire Debris: Passive Extraction of Nicotine from Components of Electronic Cigarettes Using Charcoal Strips. Poster Presentation Society of Forensic Toxicologists, Dallas TX, 2016
- McNew LA (presenter), Poklis JL, McGee Turner JB, Poklis A, <u>Peace MR.</u> A Presumptive Evaluation of Commercial Refill Formulations for Nicotine by a Microchemical Analysis Method Developed for Field Testing. Poster Presentation Society of Forensic Toxicologists, Dallas TX, 2016
- Brooks KNL (presenter), Poklis JL, McGee Turner JB, Poklis A, Peace MR. The Effects of Environment on Electronic Cigarette Eliquid Formulations. Poster Presentation Society of Forensic Toxicologists, Dallas TX, 2016
- 15. <u>Peace MR.</u> "Characterization and Abuse of Electronic Cigarettes: The Efficacy of Personal Vaporizers as an Illicit Drug Delivery System." Forensic Technology Center of Excellence Live Webinar, August 2016.
- Butler SN (presenter), Poklis JL, Turner JBM, Poklis A, <u>Peace MR.</u> "Turning Over a New Leaf: Characterization and Analysis of Kratom E-liquids." Platform presentation. Mid-Atlantic Association of Forensic Scientists, May 2016.
- 17. <u>Peace MR</u> (presenter), Poklis JL, Turner JBM, Poklis A. "Why we should care about that vapor cloud: Developing a model to study the use of e-cigarettes." Platform presentation. Mid-Atlantic Association of Forensic Scientists, May 2016.
- 18. McNew LA (presenter), Poklis JL, Turner JBM, Poklis A, <u>Peace MR</u>. "A Field Test for Nicotine in Refill Formulations for Electronic Cigarettes. Platform presentation." Mid-Atlantic Association of Forensic Scientists, May 2016.
- Mulder H (presenter), Blue IP, Patterson J, Krabowiak R, Poklis A, Poklis JL, <u>Peace MR</u>, Turner JBM. "The Hitchhiker's Guide to the Coil: A Comparison of Kanthal A-1 and Nichrome Wire in the Electronic Cigarette World." Platform presentation. Mid-Atlantic Association of Forensic Scientists, May 2016.
- Butler KE, Stone JW, Poklis JL, Turner JBM, Baird T, Poklis A, <u>Peace MR(presenter)</u>. "The Efficacy of "Personal Vaporizers" as an Illicit Drug Delivery System." Seminar. NIJ Research Symposium, February 2016.

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- Mulder HA (presenter), Blue IP, <u>Peace MR</u>, Turner JBM. "HOTWIRE: Temperature Profiling of Electronic Cigarette Coils. Poster Presentation." American Academy of Forensic Science Annual Meeting, Las Vegas, NV, February 2016.
- Butler SN (presenter), Poklis JL, Turner JBM, Poklis A, <u>Peace MR</u>. "Characterization and Analysis of Rebuildable Dripping Atomizer for Electronic Cigarettes." Poster Presentation. American Academy of Forensic Science Annual Meeting, Las Vegas, NV, February 2016.
- McLean L (presenter), Brooks K, Turner JBM, <u>Peace MR</u>. "The Risk of Electronic Cigarettes to Public Health and Criminal Justice", Poster, Society of Forensic Toxicologists Annual Meeting, Atlanta GA, October 2015.
- Stone JW (presenter), Poklis JL, Turner JBM, Poklis A, <u>Peace MR</u>. "Analysis of Marijuana E-liquid for Use in Electronic Cigarettes by Accu-TOF DART Mass Spectrometry, GC-MS, and HPLC-MS/MS/MS", Poster, Society of Forensic Toxicologists Annual Meeting, Atlanta GA, October 2015.
- Baird T (presenter), Poklis J, Stone JW, Butler KE, Wolf CE, Smith N, Turner JBM, Poklis A, <u>Peace MR</u>. "Characterization of Electronic Cigarette Refill Formulations and Dose Capture of Nicotine in Aerosol by AccuTOF DART Mass Spectrometry, HPLC-MS/MS/MS, and GC-MS", Platform, Society of Forensic Toxicologists Annual Meeting, Atlanta GA, October 2015.
- Butler KE (presenter), Poklis JL, Turner JBM, Poklis A, <u>Peace MR</u>. The Presumptive Analysis of Electronic Cigarette Aerosol Using Solid-Phase Micro-extraction (SPME) for Analysis by GCMS and AccuTOF DART-MS, Platform, Society of Forensic Toxicologists Annual Meeting, Atlanta GA, October 2015.
- 27. Murphy J, <u>Peace MR (presenter)</u>. Taking F.A.C.T.S. (Forensic Application of Critical Thinking Skills) to Middle Schools. Gulf South Summit on Service Learning and Civic Engagement through Higher Education, Little Rock, AR, March 2015.
- Baird T (presenter), Poklis J, Smith N, Turner JBM, Poklis A, <u>Peace MR</u>. "Nicotine Content in Electronic Cigarette Formulations Using Direct Analysis in Real Time (DART) AccuTOF<sup>™</sup> Mass Spectrometry and High Pressure Liquid Chromatography Triple Quadrupole Mass Spectrometry (HPLC/MS/MS)". Poster Presentation, AAFS, Orlando, February 2015.