

# Supporting information

## Cannabis Concentrate Vaping Chemistry

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Table S1

Entry	Comments	Reference
<b>Molecular composition and chemistry of vaped aerosols from concentrated cannabis (CC) samples</b>		
1	<p>Profiled total VOCs, CO and CO<sub>2</sub> derived from Δ8-THC/VEA aerosols, as well as nicotine vapes. Particle size distributions and the <i>in vitro</i> toxicity of the aerosols were studied.</p>	1
2	<p>Quantified carbonyls, organic acids, cannabinoids, and terpenes in the vaping aerosol of pure VEA, purified THC oil, and an equal volume mixture of VEA and THC oil at various coil temperatures (100-300 degrees C). It was found that degradation of VEA and cannabinoids, including Δ9-THC and cannabigerol (CBG), occurred via radical oxidation and direct thermal decomposition pathways. Evidence of terpene degradation was also observed.</p>	2
3	<p>Measured CBDQ and other select cannabinoids in nonvaped and vaped commercial CBD pure distillate oil. CBDQ was present in all commercial CBD products and significantly increased after vaping. CBDQ formed adducts with human bronchial epithelial cell proteins including Keap1 and activates KEAP1-Nrf2 stress response pathway genes.</p>	3
4	<p>Studied the mechanisms of CBD/counterfeit vape cartridge aerosol induced inflammatory response by evaluating the generation of reactive oxygen species by MCT, VEA, and cartridges and their effects on the inflammatory state of pulmonary epithelium and immune cells both <i>in vitro</i> and <i>in vivo</i>.</p>	4
5	<p>Targeted profiling of four aerosol carbonyls from EVALI patient samples. Used a commercially available sorbent bed treated with a derivatization solution to trap and stabilize reactive carbonyls in aerosol emissions to reduce reactive analyte losses</p>	5

and improve quantification. Differentiated THC versus nicotine samples. However, a broad spectrum of samples with varying liquid compositions was studied including cannabis-related active ingredients diluted in vitamin E acetate, medium chain triglycerides, squalene, and other nonpolar solvents used to develop an analytical method.

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| <b>6</b> | Focused on “vape oil liquid” heterogeneous samples and on identifying liquid and aerosol cannabinoids and terpenes. Identified > 100 terpenes and natural extracts, 19 cannabinoids, and other potential toxic additives such as vitamin E acetate, polyethylene glycols, and medium chain triglycerides. Determined more terpenes and minor cannabinoids can be produced via vaporizing and aerosolizing the vape oil. $\Delta^9$ -THC and potential toxic additives were found at lower levels in the vapor and aerosol than in the vape liquid. | 6 |
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#### **Molecular composition and chemistry of vaped aerosols from dilute cannabis samples**

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| <b>7</b> | Used diluted samples ( $\leq 10\%$ ) for vaping versus smoking toxicant exposure comparisons.  | 7 |
| <b>8</b> | CBD extracts in ethanol diluted with glycerol and vaped. During CBDA vaping most of the acidic CBDA was converted to neutral CBD and further degraded to produce several thermal products. $\Delta 8$ -iso and $\Delta 9$ -THC isomers, and cannabichromene (CBC) and CBD quinone (CBDQ) were observed from the vaping process of CBDA and CBD. In the case of $\Delta 9$ -THC psychoactive hexahydrocannabinol (HHC) derivatives and cannabinol (CBN), were produced via thermal reduction and oxidation. | 8 |
| <b>9</b> | Cannabinoids were diluted to 50 mg/mL in propylene glycol/glycerol solutions. Cannabinoids significantly increased aerosol and carbonyl formation. Most cannabinoids formed hydroxyquinones during vaping. CBD solutions produced significantly higher concentrations of formaldehyde, acetaldehyde, acrolein, diacetyl, and methylglyoxal compared with the other   | 9 |

cannabinoid samples. CBG produced higher levels of acetone, methacrolein, and methylglyoxal.

### **Profiling CC chemical compositions, liquids only, no vaping**

<b>10</b>	Edibles, <i>C. sativa</i> flower, concentrates and tinctures were analyzed in their native forms without any sample pretreatment steps prior to analysis by DART-HRMS.	10
<b>11</b>	GC-MS cannabinoid profiles obtained from vaping liquids including those from EVALI patients.	11
<b>12</b>	Studied THC levels and potencies in cannabis products.	12
<b>13</b>	Studied measurement errors in determining cannabis potency	13
<b>14</b>	Describes validation of a liquid chromatography–diode array detection (LC–DAD) method for quantification of 12 major cannabinoids in Cannabis dried plant materials, concentrates, and oils.	14
<b>15</b>	Analyses of 38 samples including initial New York cases of EVALI. The illicit fluids had relatively low cannabinoid content, sometimes with unusual $\Delta 9$ -/ $\Delta 8$ -tetrahydrocannabinol ratios, sometimes containing pesticides and many containing diluents. A notable diluent was VEA, which was found in 64% of the cannabinoid-containing fluids.	15
<b>16</b>	Of 70 e-liquids, 77.1% of the e-liquids tested underestimated or overestimated the CBD quantities stated in the product labelling.	16
<b>17</b>	Analyzed pure cannabidiol (CBD), full-spectrum, samples with CBD contents ranging from ca. 24 to 73%, and highly concentrated $\Delta 9$ - THC samples with contents of ca. 28– 69%. Density, viscosity, fusion enthalpy and melting point of CBD were determined.	17
<b>18</b>	Twenty $\Delta 8$ -THC products were purchased from retail stores in Pittsburgh, PA, USA. The range of	18

	$\Delta$ 8-THC content on independent analysis was wide and weakly correlated to the advertised content.	
<b>19</b>	Analyzed 284 e-cigarettes from EVALI patients. 82 were nicotine-containing pods, and 202 contained cannabis oil from unauthorized or black-market dealers. Cannabis cartridges had lower levels of THC and total cannabinoids, and contained significant levels of diluents including (mainly) VEA, medium-chain triglycerides, polyethylene glycol, and castor oil.	19
<b>20</b>	Studied counterfeit cartridges and identified vitamin E acetate (VEA) and tetrahydrocannabinol (THC), as well as solvent-derived hydrocarbons, silicon conjugated compounds, various terpenes, pesticides/plasticizers/polycaprolactones, and metals.	20
<b>21</b>	Measured the aromatic properties of cannabis flowers and concentrated extracts using comprehensive two-dimensional gas chromatography equipped with time-of-flight mass spectrometry, flame ionization detection, and sulfur chemiluminescence. A new family of volatile sulfur compounds were discovered as significant contributors to the cannabis scent.	21
	Found the cannabimimetic, 5F-ADB, and dextromethorphan in commercially available CBD samples.	22
<b>22</b>	A commercial $\Delta$ 8-THC distillate was found to contain eleven impurities including $\Delta$ 4,8-iso-THC, $\Delta$ 4-iso-THC, $\Delta$ 8-cis-iso-THC, 4,8-epoxy-iso-THC, 8-hydroxy-iso-THC, 9 $\beta$ hydroxyhexahydrocannabinol, 9 $\alpha$ -hydroxyhexa-hydrocannabinol, iso-tetrahydrocannabifuran, cannabicitran, olivetol, and $\Delta$ 9-THC.	23
<b>23</b>	A commercial sample of hexahydrocannabiphorol (HHCP) was found to contain impurities not associated with plant material; therefore, showing that HHCP was produced purely synthetically.	24
<b>24</b>	Among 43 disposable vape pen devices, cannabinoids such as tetrahydrocannabinol (THC) or cannabidiol (CBD) were identified in 39 of 43	25

(90.1%) samples, of which three contained both nicotine and THC.

**25** The aim of the study was to examine two commercially available electronic cigarette liquid products. Typical cannabinoids and terpenes were identified. An unknown cannabinoid was identified to be  $\Delta^9$ -tetrahydrocannabihexol acetate (Delta(9)-THCH-O. Despite products labeled to contain "60% HHCPM" and "80% 10-OH-HHC," respectively, no such compounds were detected. 26

**26** Series of studies using a variety of analytical techniques to extensive profiles of a variety of cannabinoids and derivatives in commercial products. 27, 28, 29

### **Vitamin E acetate and other diluents**

**27** Significant ROS generation by VEA vaping emissions was observed in both acellular and cellular systems. Vaped VEA caused oxidative damage and acute lung injury; the effects are more profound than exposure to equivalent concentrations of DQ alone. 30

**28** Investigated the role of the presence of molecular oxygen (O<sub>2</sub>) and transition metals in promoting thermal oxidation of e-liquids, resulting in greater degradation than predicted by pure pyrolysis. 31

**29** Investigated the emission product distribution of VEA produced at temperatures ranging from 176 to 356 degrees C. VEA degradation was found to be greatly enhanced with increasing temperature, resulting in a shift towards the production of lower molecular weight compounds, such as the redox active duroquinone (DQ) and short-chain alkenes. Low temperature vaping of VEA resulted in the production of long-chain molecules, such as phytol. 32

**30** Vaporized VEA produced 2,3,5,6-tetramethyl-1,4-benzoquinone and 2,6,10,14-tetramethyl-1- 33

pentadecene. Identification of these degradants will allow future studies to quantify and examine the degradants in vivo and in vitro as biomarkers for exposure and toxicity assessment

**31** VEA, vitamin E, coconut, and medium chain triglyceride (MCT) oil were aerosolized. The presence of some identified aerosol chemicals are known respiratory irritants and acute respiratory toxins. 34

**32** Confirmed the formation of ketene when VEA was vaped. The production of ketene increased with repeat puffs and showed a correlation to temperatures (200 to 500 °C) measured within vaping devices. Heating temperature plays an important role in ketene formation. In addition to ketene, the organic oxidant duroquinone was also obtained as another thermal degradation product of VEA. 35

**33** The objective of this study was to assess particle size distribution of VEA aerosolized using several types of vaping pens under two puffing regimens and to conduct preliminary chemical analysis of the VEA aerosol to determine if thermal degradation took place during vaping. 36

**34** A rapid method for the identification of CC additives and diluents: includes EVALI patient samples 37

#### **Indoor vaping, particle size distributions**

**35** Measured PM2.5 in the retail and consumption space of a cannabis store (a dispensary), where smoking was banned, but vaporizing and dabbing were permitted. 38

**36** Commercial cartridges containing marijuana liquid of varying CBD:THC ratios were examined. Measured exhaled aerosol within experimental rooms in two inhabited homes, collecting data on peak concentrations, decay and removal rates, and source emissions. These parameters allowed a simple exposure model to be developed. An 8-day experiment with a single puff each day showed elevated concentrations in the small experimental 39

room for the next 9 h. Mean concentrations during these hours were 63  $\mu\text{g}/\text{m}^3$ , compared to 4.5  $\mu\text{g}/\text{m}^3$  at other times.

**37** Cannabis vape liquids from nine cartridges were analyzed for several metal-bearing particles (Fe, Ni, Al, Co, Cu, Cr, V, Zn, Pb, and Sn). All samples contained a substantial number of NPs, with three samples having a number of particles one to two orders of magnitude higher than the rest of the studied samples, and the particle size distribution was, in most instances, below 150 nm for all metals. 40

**38** Metal particles in vape liquids were observed by scanning electron microscopy, and laser ablation inductively coupled plasma mass spectrometry confirmed the presence of copper-, zinc-, lead-, and manganese-bearing particles, metals that are in common alloys that may be used to make vape devices. Colocalized particles containing aluminum, silica, and sodium were also detected. These results suggest that metal particles could be a contributing factor to poor measurement precision. 41

#### **Storage and degradation chemistry**

**39** Examined emissions from cannabis smoking and vaping within an indoor environment by analyzing the gas-phase composition of the indoor air. Vaping primary indoor concentration enhancements were comparable to or higher than smoking values up to a factor of 4 among monoterpenoids. Sesquiterpenoids and some monoterpenoids showed cannabis emissions' thirdhand transport effects as concentration enhancements in the secondary indoor space. 42

**40** Thermal degradation of CBD, D9-THC, and formation of CBN was studied at 50°C, 60°C, 70°C, and 80°C for dried cannabis resin. The effect of pH and temperature on cannabinoid transformations was also examined. The major cannabinoids (CBD, CBN, and  $\Delta$ 9-THC) reacted relatively quickly at high temperature and in an acidic solution. Onset of degradation of CBD, CBN, and  $\Delta$ 9-THC was 43

achieved by using low temperature, slightly to moderately acidic pH values, and short-time processing.

- 41** The results of this study indicate that levels of CBD, THC, and CBN within vape liquids consumers purchase can change dramatically depending on the storage conditions (time and temperature) of the vape liquids. 44

#### **Device Parameters**

- 42** Infrared thermography was used to measure heating coil temperatures in one temperature-controlled and two voltage-controlled systems. The cartridges were modified for direct line-of-sight on the heating coils, the wick and coils were saturated with cannabis extract, and fixtures were developed to force two liters per minute air flow past the coils for the full duration of the puff allowed by the device. Voltage alone was a poor indicator of coil temperature and only the temperature-controlled system consistently maintained temperatures less than 400 degrees C for the full puff duration. 45

- 43** Investigated the composition and internal components of 2019 EVALI patient THC and nicotine devices from 2016–19, specifically the metal, ceramic, and polymer components likely to be exposed to heat by disassembling forty-eight components from eight used and unused vaping devices under a microscope and analyzed them using X-ray fluorescence, scanning electron microscopy, and Fourier-transform infrared micro-spectroscopy. The combination of ceramics, metals, and high temperatures in newer THC cartridges is consistent with conditions hypothesized to produce VEA reactions during vaping. 46

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