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Comparison of select analytes in aerosol from e-cigarettes with smoke from conventional cigarettes and with ambient air

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ABSTRACT

Leading commercial electronic cigarettes were tested to determine bulk composition. The e-cigarettes and conventional cigarettes were evaluated using machine-puffing to compare nicotine delivery and relative yields of chemical constituents. The e-liquids tested were found to contain humectants, glycerin and/or propylene glycol, (\geq 75% content); water (<20%); nicotine (approximately 2%); and flavor (<10%). The aerosol collected mass (ACM) of the e-cigarette samples was similar in composition to the e-liquids. Aerosol nicotine for the e-cigarette samples was 85% lower than nicotine yield for the conventional cigarettes. Analysis of the smoke from conventional cigarettes showed that the mainstream cigarette smoke delivered approximately 1500 times more harmful and potentially harmful constituents (HPHCs) tested when compared to e-cigarette aerosol or to puffing room air. The deliveries of HPHCs tested for these e-cigarettes; no significant contribution of cigarette smoke HPHCs from any of the compound classes tested was found for the e-cigarettes. Thus, the results of this study support previous researchers' discussion of e-cigarette products' potential for reduced exposure compared to cCBY license (http://

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1. Introduction

Electronic cigarettes (e-cigarettes) are a relatively new consumer product. Unlike conventional cigarettes, e-cigarettes do not burn tobacco to deliver flavor. Instead, they contain a liquidbased flavorant (typically referred to as e-liquid or e-juice) that is thermally vaporized by an electric element. This liquid typically consists of a mixture of water, glycerin, and/or propylene glycol. The liquid also contains nicotine and flavor, although nicotine-free products are available.

While there are decades of characterization studies and numerous standardized analytical procedures for conventional cigarettes,

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relatively little published analytical data exists for commercial ecigarette products. Furthermore, no standardized test methods or reference products exist for e-cigarettes.

Electronic cigarettes are generally purported to provide reduced exposure to conventional cigarettes' chemical constituents because they deliver flavors and nicotine through vaporization rather than by burning tobacco. Goniewicz et al. (2014) reported low levels of select chemical constituents in select e-cigarette brands commercially available in Poland. A recent review of analyses from diverse e-cigarettes shows comparatively simple chemical composition relative to conventional cigarette smoke (Burstyn, 2014). However, limited published results exist for commercial products that represent a significant presence in the marketplace (Cheng, 2014).

The purpose of this study was to evaluate e-cigarette products with a significant presence in the marketplace for bulk composition, including nicotine, and for select constituents for comparison with conventional cigarette products. Three blu eCigs products (approximately 50% of the US market) and two SKYCIG products (approximately 30% of the UK market) were chosen for evaluation. Marlboro Gold Box (US), and Lambert & Butler Original and Menthol products (UK), with significant market share in their respective geographical areas, were included in the study for conventional cigarette comparisons.

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Abbreviations: ACM, aerosol collected mass; HPHC, harmful and potentially harmful constituents; CO, carbon monoxide; TSNA, tobacco-specific nitrosamines; PAA, polyaromatic amines; PAH, polyaromatic hydrocarbons; LOQ, limit of quantitation; LOD, limit of detection; CAN, Health Canada Test Method T-115; blu CTD, Classic Tobacco Disposable; blu MMD, Magnificent Menthol Disposable; blu CCH, Cherry Crush, Premium, High Strength; SKYCIG CTB, Classic Tobacco Bold; SKYCIG CMB, Crown Menthol Bold; MGB, Marlboro Gold Box; L&B O, Lambert & Butler Original; L&B M, Lambert & Butler Menthol; TPM, total particulate matter; PG, propylene glycol.

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The products used in the study were evaluated for content and delivery of major ingredients (glycerin, propylene glycol, water, and nicotine) and for select constituents (carbon monoxide (CO), carbonyls, phenolics, volatile organic compounds (volatiles), metals, tobacco-specific nitrosamines (TSNAs), polyaromatic amines (PAAs), and polyaromatic hydrocarbons (PAHs)). Many of these constituents are included in cigarette industry guidance issued by the FDA that includes reporting obligations for harmful and potentially harmful constituents (HPHCs) in cigarette filler and smoke under section 904(a)(3) of the 2009 Family Smoking Prevention and Tobacco Control Act (FDA, 2012). For delivery studies, the conventional cigarettes were smoked under an intense puffing regime published by Health Canada (1999). The e-cigarettes were tested using minimal modifications to this smoking regime. Ninety-nine puffs were used to collect approximately the same aerosol mass as obtained from conventional cigarette testing. Ambient 'air' samples, empty port collections, were included as a negative control of aerosol testing for cigarette constituents (i.e. HPHC).

2. Materials and methods

2.1. Test products

Two disposable e-cigarette products and three rechargeable ecigarette products were obtained from the manufacturers. Three conventional cigarette products were purchased through wholesale or retail sources for testing. Information for each of the products is listed in Table 1.

2.2. Methods overview

ISO 17025 accredited analytical methods were used to evaluate the cigarette samples for select HPHCs in mainstream smoke. Official methods are cited and other, internally validated, methods are briefly described for general understanding. Furthermore, because no standardized methods exist for e-cigarette analysis, the methods used to evaluate the conventional cigarettes were adapted to evaluate the e-cigarette products and the study blanks (room air). In an effort to maximize signal and lower methods' limits of quantitation, aerosol collection amounts were maximized (but maintained below breakthrough) and extraction solvent volumes were minimized. In some cases, alternative instrumentation was employed to improve detection. For example, mainstream smoke TSNAs were analyzed by GC-TEA while aerosol and air blank samples were analyzed by LC-MS/MS. Accuracy, precision, and method limits of quantitation and detection (LOQ and LOD) were verified for each method. On average, accuracy and method variability for the analytes tested were determined to be 98% and 3%, respectively. Analyte LOD and LOQ information is listed in Supplemental Appendix A Tables 1 and 2. Method resolution for low levels of analytes was influenced by background levels of select analytes in air control samples. These background levels are attributed to

Table 1

List of cigarette and e-cigarette products tested.

| Product | Manufacturer | Product type | Nicotine information provided on packaging |
|--|-------------------|--------------------------|--|
| Classic Tobacco Disposable (blu CTD) | blu eCigs | Disposable e-cigarette | Content: 24 mg/unit |
| Magnificent Menthol Disposable (blu MMD) | blu eCigs | Disposable e-cigarette | Content: 24 mg/unit |
| Cherry Crush, Premium, High Strength (blu CCH) | blu eCigs | Rechargeable e-cigarette | Content: 16 mg/unit |
| Classic Tobacco Bold (SKYCIG CTB) | SKYCIG | Rechargeable e-cigarette | Content: 18 mg/unit |
| Crown Menthol Bold (SKYCIG CMB) | SKYCIG | Rechargeable e-cigarette | Content: 18 mg/unit |
| Marlboro Gold Box (MGB) | Philip Morris USA | Conventional cigarette | - |
| Lambert & Butler Original (L&B O) | Imperial Tobacco | Conventional cigarette | Yield: 0.9 mg/cig (ISO) |
| Lambert & Butler Menthol (L&B M) | Imperial Tobacco | Conventional cigarette | Yield: 0.5 mg/cig (ISO) |

instrument or smoking machine carry-over as evidenced in solvent or air blanks. In addition, the high concentration of glycerin and water in e-cigarette aerosol present challenges for volatile-based measurement systems (i.e. GC). Additional method refinements and dedicated e-cigarette puffing machines are two areas for consideration to improve e-cigarette aerosol method sensitivities. Method development and verification details for e-cigarette liquids and aerosols are the subject of a future publication.

2.3. Smoke and aerosol collection

Cigarette preparation and machine smoking for conventional cigarettes are described in Health Canada Test Method T-115 (CAN) (1999). Two to three cigarettes were smoked per replicate for conventional cigarettes and 99 puffs were taken from single e-cigarettes for no more than approximately 200 mg of particulates collected per pad. Three to five replicates were tested for each measurement. Prior to analysis, filter pads from cigarette smoke collection were visually inspected for overloading of particulates, as evidenced by brown spotting on the back of the filter pad. To ensure no overloading of particulates for aerosol collection. e-cigarette units were weighed before and after collection to verify that product weight change and filter pad weight change were comparable. Air blanks were prepared by puffing room air (99 puffs) through an empty smoking machine port to the indicated trapping media for an analysis method. These air blank samples were prepared and analyzed in the same manner and at the same time as the e-cigarette aerosol samples. Smoke and aerosol collection sections were conducted separately. Smoke and aerosol particulate was collected onto 44 mm glass fiber filter pads with >99% particulate trapping efficiency for each replicate analysis. For carbonyls, smoke/aerosol was collected directly by two impingers, in series. For smoke metals analysis, electrostatic precipitation was used. For volatiles and PAH determinations, single chilled impingers were placed in-line with the filter pads. e-Liquid glycerin and nicotine were quantitated using GC-FID and/or GC-MS using a method equivalent to ISO 10315 (ISO, 2000a). e-Liquid water was quantitated using Karl Fischer analysis. A reference e-liquid was developed and used as a testing monitor for ingredient determinations in the e-liquid samples. The reference e-liquid is composed primarily of glycerin, propylene glycol, and water with low levels of nicotine, menthol, and Tween 80. The Tween 80 is added to improve solubility of menthol in the solution. The reference is not meant to directly mimic an e-liquid used for consumption but merely used for analytical control charts. Three replicates were tested for each sample and the reference.

2.4. Analytical assays

Carbon monoxide was determined concurrently with aerosol and smoke collection for nicotine and water and analyzed by NDIR using ISO method 8454:2007 (ISO, 2007). Carbonyls were trapped using 2,4-dinitrophenylhydrazine as a derivatizing agent with

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