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iQOS: Evidence of Pyrolysis and Release of a Toxicant from Plastic

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#### ABSTRACT

**Objective:** To evaluate performance of the iQOS Heat-Not-Burn system as a function of cleaning and puffing topography, investigate the validity of manufacturer's claims that this device does not burn tobacco, and determine if the polymer-film filter is potentially harmful. **Methods:** iQOS performance was evaluated using five running conditions incorporating two different cleaning protocols. Heatsticks were visually and stereomicroscopically inspected preand post- use to determine the extent of tobacco plug charring (from pyrolysis) and polymer-film filter melting, and to elucidate the effects of cleaning on charring. GC-MS headspace analysis was conducted on unused polymer-film filters to determine if potentially toxic chemicals are emitted from the filter during heating.

**Results:** For all testing protocols, pressure drop decreased as puff number increased. Changes in testing protocols did not affect aerosol density. Charring due to pyrolysis (a form of organic matter thermochemical decomposition) was observed in the tobacco plug after use. When the manufacturers' cleaning instructions were followed, both charring of the tobacco plug and melting of the polymer-film filter increased. Headspace analysis of the polymer-film filter revealed the release of formaldehyde cyanohydrin at 90°C, which is well below the maximum temperature reached during normal usage.

**Discussion:** Device usage limitations may contribute to decreases in interpuff intervals, potentially increasing user's intake of nicotine and other harmful chemicals. This study found that the tobacco plug does char and that charring increases when the device is not cleaned between heatsticks. Release of formaldehyde cyanohydrin is a concern as it is highly toxic at very low concentrations.

#### INTRODUCTION

With the rise of smoking alternatives, the ENDS (electronic nicotine delivery systems) market has boomed, with electronic cigarettes (EC) being among the most popular worldwide <sup>12</sup>. However, there are still a number of conventional (combustible) cigarette smokers who would welcome a cigarette-like tobacco/nicotine containing product that is devoid of or has a significantly reduced toxicity compared to conventional cigarettes<sup>1</sup>. To appeal to this demographic, Phillip Morris International has released a new product called the iQOS (I quit original smoking), which is a "heat-not-burn" system <sup>3</sup>, as an alternative to conventional cigarettes and EC. The iQOS system uses a flange, called the "Heater", which is composed of a silver, gold, platinum, ceramic coating <sup>4</sup>, to heat a rolled, cast-leaf sheet of tobacco impregnated with glycerin, thereby creating an aerosol without combustion<sup>3</sup>. This aerosolization process is proposed to reduce a user's exposure to toxic and carcinogenic chemicals produced by the combustion of tobacco <sup>5</sup> <sup>6</sup>. Thus, the consumer gets the "harm reduction" component of EC along with the mouth/throat feel of a conventional cigarette. The iQOS system has been well received in Japan and Italy. The iQOS is currently sold in 26 markets by PMI with plans to expand to over 30 countries, including the US<sup>7</sup>.

Although this product has been extensively evaluated by the manufacturer <sup>3 5 6 8 9 10 11 12 13</sup>, these studies appeared in a journal that may have a deficient review process <sup>14</sup>, emphasizing the need for independent evaluation of the iQOS. As our initial study, we have evaluated the performance of the iQOS system under various conditions, tested the effects of cleaning on performance and pyrolysis, and determined the composition of and potential health risk from the polymer-film filter.

#### **MATERIALS & METHODS:**

#### iQOS Product Acquisition and Storage

Four iQOS tobacco heating system kits, manufactured by Phillip Morris Products S.A. (Switzerland), were purchased online at Ebay.com from sellers with a 98% or higher satisfaction rating. Kits arrived sealed and in excellent condition. Kits were inventoried, and the components of each kit were placed into individual plastic containers and stored in a dry area at 22°C when not in use.

Cartons of Marlboro (blue box) heatsticks, manufactured by Phillip Morris Brands Sàrl (Italy), were purchased in Japan and shipped to us via a personal shopper. Each carton was individually sealed and in excellent condition. Heatsticks were stored, unopened, in a dry, dark area at 22°C in their cartons until used. Unused heatsticks from opened packs were stored in an airtight bag in their carton.

#### Cleaning the iQOS

iQOS Holders were tested using two cleaning regimens: (1) the "Per Use" cleaning protocol in which the device was thoroughly cleaned after each heatstick using the cleaning sticks to remove residual fluid and tobacco plug debris from the Heater and surrounding base and to clean out the cap; and (2) the manufacturer's recommended cleaning instructions in which the cleaning cycle was used after every 20 heatsticks before using the brush cleaners. When heatstick fragments were left behind, the cleaning hook was used to remove these pieces, and as necessary, the Holder Cap was cleaned by a 5-minute warm water emersion. The instructions clearly state that the Holder itself is not to be wetted.

#### **Performance Evaluation**

Pressure drop, which measures the draw resistance of the heatstick, aerosol absorbance (density), a measure of particulate matter trapped within the aerosol, and puff number were evaluated for iQOS products using equipment and protocols described previously <sup>15 16 17</sup>. Pressure drop across heatsticks was evaluated using a Cole-Palmer Masterflex L/S peristatic pump (Vernon Hills, IL) connected to a U-tube water monometer to detect the change in differential pressure for each puff. Airflow rates were pre-calculated/calibrated to the appropriate pump speed using a conversion factor provided by the pump head manufacturer, and flow rate was verified using a Brooks Instruments Sho-Rate flow meter (Hatfield, PA). Aerosol density was evaluated by capturing aerosols in a tubular cuvette, and absorbance was measured immediately at 420 nm using a Bausch & Lomb spectrophotometer (120 Volts, 0.9 Amps, Rochester, NY).

iQOS devices were evaluated with five operating conditions; four (#s 1-4) used the Per Use cleaning protocol and one (#5) used the manufacturer's recommended cleaning instructions. The pump head, tubing set-up and running conditions were as follows: (1) Low Airflow Rate 2 Second Protocol - the peristaltic pump was outfitted with a Cole-Palmer Masterflex Model 7015-21 pump head (standard pump head) utilizing Masterflex Tygon E-LFL (Tubing Size 15) tubing to generate a flow rate of 7mL/sec with a 2 second puff duration for a total puff volume of 14 mL, 14 puffs were taken at 25 second intervals; (2) Low Airflow Rate 4 Second Protocol - the same pump set-up and running conditions as for condition #1 with a 4 second puff duration generating a 28 mL puff volume; (3) International Organization of Standardization standard (ISO) - the pump was outfitted with a Cole-Palmer Masterflex L/S Easy-Load II Model 77200-52 high performance pump head with Masterflex Tygon E-LFL (Tubing Size 15) producing a 17.5mL/sec flow rate with a 2 second puff duration, generating a total puff volume of 35 mL, with a total of 6 puffs taken, one puff every minute; (4) the Health Canada standard (HCI) - a Cole-Palmer Masterflex L/S Easy-Load II Model 77200-52 high performance pump head was used with Masterflex Tygon E3603 (Tubing Size 36) tubing for a flow rate of 27.5mL/sec, with a 2 second puff for a total puff volume of 55 mL, 12 puffs were taken at 30 second intervals; (5) Manufacturer's Recommended Cleaning (HCI), the same pump set-up and running conditions as described for condition #4 but in the absence of Per Use cleaning; for this protocol the manufacturer's recommended cleaning instructions were followed (Table 1). For conditions #s 1-4, three different iQOS devices were evaluated with each device being tested in triplicate, i.e. a new heatstick was used for each experiment; #5 employed a single device in which 10 heatsticks were tested without cleaning between each stick.

#### Table 1. Performance of iQOS Heat-Not-burn Holders

Holder	Puff Duration)	Puff Interval	Air- flow Rate (mL/s)	Puff Volume (mL)	Total # of Puffs	Average Pressure Drop (mmH <sub>2</sub> O)	Average Absorbance
Low Airflow Rate 2 Second Protocol <sup>a</sup>							
А	2	25	7	14	14	$13 \pm 5$	$0.42\pm0.08$
В	2	25	7	14	14	$13 \pm 4$	$0.45\pm0.08$
С	2	25	7	14	14	$18 \pm 7$	$0.46\pm0.06$
Low Airflow Rate 4 Second Protocol <sup>b</sup>							
А	4	25	7	28	14	$9\pm4$	$0.41 \pm 0.05$
В	4	25	7	28	14	$11 \pm 4$	$0.46\pm0.09$
С	4	25	7	28	14	$10 \pm 4$	$0.49\pm0.04$
ISO Standard <sup>c</sup>							
А	2	60	17.5	35	6	$62 \pm 5$	$0.49\pm0.10$
В	2	60	17.5	35	6	$65 \pm 8$	$0.54 \pm 0.09$
С	2	60	17.5	35	6	$57 \pm 5$	$0.49\pm0.04$
HCI Standard <sup>d</sup>							
А	2	30	27.5	55	12	$103 \pm 9$	$0.26 \pm 0.03$
В	2	30	27.5	55	12	$100 \pm 9$	$0.41 \pm 0.05$
С	2	30	27.5	55	12	$105 \pm 13$	$0.42 \pm 0.05$
Manufacturer's Recommended Cleaning (HCI) <sup>e</sup>							
Е	2	30	27.5	55	12	$103 \pm 12$	$0.46 \pm 0.06$
Note <sup>a</sup> Per use cleaning protocol standard nump head with Tygon 15 tubing							

*Note.* <sup>a</sup> Per use cleaning protocol, standard pump head with Tygon 15 tubing. <sup>b</sup> Per use cleaning protocol, standard pump head with Tygon 15 tubing. <sup>c</sup> Per use cleaning protocol, high performance pump head with Tygon 15 tubing.

<sup>d</sup> Per use cleaning protocol, high performance pump head with Tygon 36 tubing.

<sup>e</sup> Manufacturer's recommended cleaning, high performance pump head with Tygon 36 tubing.

#### Effect of Use on the Tobacco Plug and Polymer-film Filter

The condition of the tobacco plugs was evaluated by visual and microscopic inspection and imaged using a Nikon C-LEDS stereomicroscope equipped with a Nikon Digital Sight DS-Vi1 camera head (Nikon, Minato, Tokyo, Japan) before and after use. Some heatsticks were dissected before and after use to further evaluate residual char (referred to as "char" only) of the tobacco plugs and the condition of the polymer-film filter.

#### GC-MS Analysis of iQOS Heatstick Polymer-Film Filters

Gas chromatography-mass spectrometry (GC-MS) utilizing a qualitative wide-scope screening method was performed using an Agilent 7890B GC coupled with a 5977A MSD equipped with a 7698A Headspace Sampler (Santa Clara, CA). Evaluation of iQOS aerosols was performed using headspace analysis. Chromatographic separation was accomplished using an Agilent J&W HP-5ms Ultra Inert GC Column (30 m x 0.25 mm x 0.25 µM) (Santa Clara, CA) and ultra-pure helium (>99.999% purity) as the carrier gas at a flow rate of 1.5 mL/min. For headspace analysis, three unused heatsticks were dissected, polymer-film filters were removed, and 3 mm of the portion (16.7%) closest to the tobacco plug were excised and placed into 20 mL head space vials. All samples were analyzed with a split ratio of 50:1, a solvent delay of 2 min, with blank analysis between each sample. GC ramp conditions were as follows: 40 °C for 5 min, 45 °C for 5 min, 90 °C for 5 min, 130 °C for 5 min, 135 °C for 5 min, 165 °C for 5 min, 190 °C for 2 min, all temperature ramps were at 10 °C/min. Ionization of compounds was performed using electron impact ionization at 70 eV in positive mode, the ion source maintained at 250 °C and chemicals were identified using the NIST mass spectral library (Gaithersburg, MD), only chemicals with an 85% or higher probably match were listed as identifiable.

#### RESULTS

#### **Components in the iQOS Heat-Not-Burn System**

The iQOS Kit (figure 1 A-G) consisted of an instruction manual written in German, English, Portuguese, and Italian, a Pocket Charger, the Holder (device), a USB cable and a European wall adapter plug for charging, moist cleaning sticks to clean the Holder and cap, and the cleaner, which contains a long brush for cleaning the inside of the Holder, where the Heater is housed, a short brush for cleaning the Cap, and a hook for removing pieces of tobacco plug left in the Holder/Cap. A universal power adapter was purchased from Amazon.com and used to charge the Pocket Charger unit. Each carton of iQOS heatsticks contained 10 individually wrapped packs, and each pack had 20 heatsticks (figure 1H).

The iQOS kit components had an overall feel of good craftsmanship. The fabrication of the tobacco plug cast-leaf demonstrates a waste not want not strategy in that the plug is fabricated from pulverized tobacco remnants/waste materials, including tobacco stems, torn leaf material, and leaf dust <sup>18</sup>. These items are reconstituted with natural adhesives and glycerin (a solvent that is used in EC fluids to produce aerosol) and processed into sheets forming cast-leaf, which is rolled and used as the tobacco plug <sup>3</sup>.

#### **Cleaning of iQOS Device**

The interior chamber of the Holder contained a heating element, referred to in the iQOS instruction manual as the Silver Gold Platinum Ceramic Coated Heater (figure 2). Unused Holders were clean and debris-free with a white base and white Heater with a metallic coil in its center (figures 2A-C). Used Holders that were thoroughly cleaned with the cleaning sticks between each heatstick were generally similar to the unused Holder, except that the heating

element had deposits of hardened dark debris that was not removed by the cleaning stick, cleaning cycle of the Pocket Charger, or long brush (figure 2 D-F). In the used Holder that was not cleaned between heatsticks (manufacturers recommend cleaning), brown liquid and particulates covered the base, walls and Heater (figure 2G-I). With continued use in the absence of cleaning, the volume of liquid and debris increased, and the pieces of debris became darker and appeared more charred (figure 2 D-I were taken after the 10<sup>th</sup> heatstick was used).

#### iQOS Performance

The iQOS gives users a maximum of 14 puffs during a 6-minute window per heatstick, after which it must be recharged before it can be used again. Performance of the iQOS was evaluated using five puffing protocols (figure 3, Table 1). For protocols 1-4, three different iQOS devices (Holder A, B, and C) were tested in triplicate, i.e. a new heatstick was used for each experiment, and each device underwent an intensive cleaning between each heatstick. For protocol 5, a single device (Holder E) was used, and it was not cleaned between 10 heatsticks (average of the first three heatsticks is shown in figure 3I, J). For all testing protocols, pressure drop decreased as puff number increased. Aerosol density readings increased with use, peaking around puff 7- 9, and then begin to decrease. Although pump set-up affected pressure drop, it did not affect aerosol absorbance which remained similar under all running conditions, However, differences in testing conditions may lead to alterations in the chemical constituents present within the aerosol without altering aerosol density. Not-cleaning did not affect performance except that pressure drop was more variable during the first 4 puffs in the uncleaned trials.

#### **Tobacco Plug Charring**

Dissection of unused and used heatsticks showed tobacco plug charring (figure 4A). Stereomicroscopic comparison of unused (figure 4B) and used (figure 4C) tobacco plugs confirmed charring or blackening of the cast-leaf. Visual and stereomicroscopic inspection of used heatsticks show the effects cleaning had on device heat production. Comparison of the first and tenth used heatstick from Holder A (Per-use cleaning) shows that with regular cleaning the charred area surrounding the Heater, referred to as the zone of charring, does not increase with use (figure D-E). The effects of cleaning on heating were most evident during the course of the Manufacturer's Recommended Cleaning (HCL) testing. Comparison of these heatsticks to unused and Per-use cleaned heatsticks showed that in the absence of regular cleaning, the zone of charring increased as the number of heatsticks tested increased (figure 4H-L).

#### **Polymer-Film Melting**

Effects of cleaning on heating were not exclusive to the tobacco plug; figure 4A shows that the polymer-film filter (#2), which is separated from the tobacco plug (#4) by the hollow acetate filter (#3), was adversely effected. The aerosol produced by the iQOS was hot enough to melt the polymer-film filter, which could allow release of potentially hazardous chemicals. Melting of the polymer-film filter was evident by slight yellowing of the filter, as well as by narrowing of the end closest to the tobacco plug (figure 4A indicated by black arrow). This melting and subsequent cooling of the filter caused it to harden, preventing it from being longitudinally dissected. Comparison of unused and used polymer-film filters from both Per-use and Manufacturer's Recommended Cleaning experiments showed the relationship between cleaning and increased heat generation. First (figure 4F) and tenth (figure 4G) filters from

cleaned devices showed similar discoloration and melting to that of the first filter from the uncleaned device (figure 4N). Comparison of these heatsticks to subsequent Manufacturer's Recommended Cleaning used heatsticks showed discoloration and melting of the polymer-film filter increased with increased use (figures 4M-Q).

#### Head Space analysis of unused polymer-film filters

GC-MS headspace analysis of unused polymer-film filters showed the presence of  $\varepsilon$ caprolactone and lactide, common components in plastics, as well as 1,2-diacetin, a plasticizer (figure 4R). However, of most concern was the presence of formaldehyde cyanohydrin (glycolonitrile), an acute toxicant often used in the production of synthetic resins and used as a solvent <sup>19</sup>. Formaldehyde cyanohydrin was eluted at 17.97 minutes, when the column reached 90°C.

#### DISCUSSION

Unlike some EC, which often show significant variation in craftsmanship and performance within and between brands <sup>15 20</sup>, the iQOS appearance, design, and performance data are consistent with a product that is well manufactured. However, some design features of the iQOS, such the limited time allowed per heat stick and the need to consume the entire heat stick within this time or alternatively waste part of it, will affect user's topography and may lead to unwanted exposure to potentially toxic chemicals emitted from melting plastic and from pyrolysis of tobacco.

In contrast to tobacco and EC, which usually have no constraints on puffing, the iQOS only operates for 6 minutes, at which time it automatically shuts off and requires charging before it can be used again. Since a maximum of 14 puffs can be taken from each iQOS heatstick, puffing needs to be done at about 25 second intervals to take full advantage of each heatstick; used heatsticks that have not been fully exhausted cannot be used again as reinsertion would cause the delicate cast-leaf tobacco plug to crumble. This may not appeal to all users, and users who puff less frequently would have a lower number of puffs/heatstick. For users wishing to maximize each heatstick, this limitation will force them to alter their smoking topography by decreasing the interpuff interval and/or accelerating the rate at which they puff, leading to larger volumes of aerosol inhalation.

The manufacturer's cleaning instructions were not fully developed in the instruction manual. The cleaning protocol recommended using the cleaning function of the charger followed by cleaning with the brushes after 20 heatsticks and removing any large fragments of tobacco plug with the Hook if necessary. The iQOS kit was equipped with cleaning sticks (figure 1B and D), yet their use was not mentioned in the instruction manual. Our data show that use of one heatstick left a significant amount of debris, fluid, and fragments of cast-leaf in the Holder (figure 2).

While iQOS heatsticks do not produce a flame, they were always charred after use, which we interpret to be a result of pyrolysis. The zone of charring was greater when cleaning was not performed between heatsticks, suggesting that buildup of fluid and debris in the Holder increases pyrolytic temperatures. These data are consistent with the idea that despite similarities in performance characteristics, the cleanliness of the device plays a critical role in thermal regulation. Pyrolysis of tobacco is an endothermic reaction which occurs at temperatures between 200 and 600 °C, during which the majority of volatile and semi-volatile components of cigarette smoke are formed <sup>21,22</sup>. Although the Phillip Morris study indicated that the aerosol produced by iQOS devices reduce the amount of chemicals found on the FDA's Harmful and Potentially Harmful Constituents list by limiting tobacco pyrolysis <sup>5</sup>, our study, showing charring, in conjunction with a study by Auer et al., which confirmed the presence of volatile organic compounds, polycyclic aromatic hydrocarbons, carbon dioxide, and nitric oxide <sup>23</sup>, contradict the claim that tobacco pyrolysis is minimized in iQOS. Although iQOS operates at temperatures less than 350°C, this does not negate the formation of volatile and semi-volatile harmful constituents of tobacco smoke, which tend to have boiling points that range from 70 to 300°C<sup>21,22</sup>.

Heatsticks used in this experiment were dissected and the severity of polymer-film filter melting was examined. The function of the polymer-film filter is to cool the aerosol <sup>3</sup>, thus it would seem that the polymer composing the film should be heat resistant, although,  $\varepsilon$ -caprolactone, also known as polycaprolactone or PCL, tends to have low melting point which is thickness dependent <sup>24</sup>. The intensity of the heat produced by the iQOS, under both cleaned and

uncleaned conditions, was sufficient to melt the polymer-film filter, even though it was not in direct contact with the Heater. The amount of damage to the film (increase in melt and alteration of coloration) increased with each heatstick when cleaning was done per the manufacturer's recommended procedure (after 20 heatsticks). Discoloration may be a product of heating and/or staining from the brown fluid that is expelled from the tobacco plug during use.

Our GC-MS data indicate that components of the polymer-film filter are aerosolized at relatively low temperatures. GC-MS headspace analysis of unused filters suggests the polymer-film filter is a combination of ε-caprolactone, lactide, 1,2-diacetin and other unidentified chemicals. The chemicals released from the film filter during heating may not be suitable for inhalation. Thus, it is unknown if the film filter material is safe for use in products where it would undergo intense cycles of heating and cooling. Of greatest concern was the release from the polymer filter of formaldehyde cyanohydrin, a highly toxic chemical that is metabolized in the liver and broken down into formaldehyde and cyanide <sup>19</sup>. Formaldehyde cyanohydrin can be fatal to humans <sup>19 25 26</sup>, with studies showing mouse inhalation LD<sub>Lo</sub>, the lowest dose of a toxicant that causes the death of an animal <sup>27</sup>, values as 27 ppm/8 hr <sup>28 29</sup>. iQOS Holders operate at temperatures between 330-349°C <sup>3 23</sup>, and as a safety feature, the device shuts off when temperatures reach 350°C. The release of formaldehyde cyanohydrin from unused filters during GC-MS analysis occurred at 90°C, a temperature that all users will exceed.

In conclusion, the iQOS appears to be well manufactured, and performance data were consistent between heatsticks. However, the product has limitations that will affect user topography and the application of standard smoking protocols, such as the ISO 3308, which could not be used for more than six puffs with this product. Users may be forced to smoke at a rapid pace in order to fully maximize heatsticks. Decreasing the interpuff interval could lead to an increase in intake of nicotine <sup>30</sup> and carbonyls <sup>31</sup>. This study also showed that the iQOS is not strictly a "heat-not-burn" tobacco product. The iQOS tobacco appeared to char without ignition, and charring increased when cleaning was not done after each use. This study also showed the potential dangers that the polymer-film filter poses. This thin plastic sheet, readily melts during iQOS use and releases formaldehyde cyanohydrin, a dangerous toxicant. This study has shown that the iQOS system may not be as harm-free as claimed and also emphasizes the urgent need for further safety testing as the popularity and user base of this product is growing rapidly.

#### WHAT THIS PAPER ADDS

Performance characteristics were generally uniform between devices and heatsticks.

iQOS device usage limitations make modifications to some current smoking standards necessary for proper evaluation of products.

Device limitations may decrease users' interpuff intervals, increasing possible toxic exposures.

iQOS Holders heat hot enough to cause charring of the tobacco plug via pyrolysis and melting of the polymer-film filter.

iQOS Holder cleanliness affects and contributes to increased charring of the tobacco plug and melting of the polymer-film filter.

Formaldehyde cyanohydrin, a toxicant, was released from the polymer-film filter at 90°C

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#### **COMPETING INTERESTS None**

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**Figure 1.** The iQOS heat-not-burn system. **(A)** An iQOS starter kit. **(B)** The kit consists of an instruction manual, iQOS Pocket Charger, iQOS Holder, USB cable, iQOS Cleaning Sticks, wall charging adapter, and iQOS Cleaner. **(C)** Profile view of iQOS holder inside a Pocket Charger. **(D)** Individual pack of iQOS Cleaning Sticks with an example of an unused stick and a stick after a single use per end. **(E)** A closed and opened iQOS Cleaner; the larger end contains the long brush and protruding cleaning hook, and the shorter end contains the short brush. **(F)** Internal view of the iQOS cleaner showing the two brushes (long brush on the left, short brush on the right). **(G)** The Cleaning Hook removed from the iQOS Cleaner. **(H)** Marlboro iQOS Heat Stick carton (containing 10 individual packs), sealed individual pack, and opened pack exposing heat sticks.

**Figure 2.** Internal view of the iQOS Holder. **(A-C)** Clean, unused Holder showing heater (blue arrows), **(D-F)** Used Holder that was cleaned after every use; black residue remains on heater (red arrows), **(G-I)** Used Holder that has not been cleaned between uses (10 uses).

**Figure 3.** Performance characteristics of the iQOS heat- not-burn system. **(A, C, E, G, and I)** Pressure drop is plotted versus the puff number for five puffing protocols. **(B, D, F, H, and J)** Absorbance is plotted versus puff number for the five puffing protocols. Each line of the graphs represents the average of three heatsticks for an individual Holder (Holder A = red, Holder B = green, Holder C = blue, and Holder E = Purple).

**Figure 4. Charring of Tobacco Plug and Melting of Polymer-film Filter. (A)** Dissected Heat Sticks, each Heat Stick is composed of: (1) the low-density cellulose mouth piece filter, (2)

polymer-film filter, (3) hollow acetate tube, and (4) tobacco plug. Heat Sticks from left to right are unused stick with the paper over-wrap peeled away, and used stick with the paper over-wrap removed with the mouthpiece filter and hollow acetate tube sliced open; black arrow indicates melted region of the polymer-film filter, black asterisks denote tobacco plug fragments that have been drawn into the hollow acetate tube. (B) An unused tobacco plug. (C) Used tobacco plug showing charring/ darkening with use. (D-E) Cross sections of tobacco plugs from the first (D) and tenth (E) heat stick of Holder A of the cleaned experiment. CHC = Cleaned Device. Yellow outlined area indicates a void in the cast leaf left by the Heater, the area between the yellow and green outlines are the charred portions of the tobacco plug. (F-G) Cross sections of polymer-film filter from the first (F) and tenth (G) heat stick. Polymer-film filter images shown coincide with tobacco plug images D-E. (For D-G, CHC = Cleaned Device Heatstick). (H-L) Cross sections of tobacco plugs before use (H) and after use from the first, fourth, sixth, and tenth heat stick of the uncleaned experiment (I-L). Yellow outlined area = void in the cast leaf left by the Heater, area between the yellow and green outlines = charred portions of the tobacco plug. (M-Q) Cross sections of polymer-film filter before (M) and after use (N-Q). Polymer-film filter images shown coincide with tobacco plug images H-L. Blue arrowheads show charred pieces of tobacco plug that are affixed to the tobacco plug (K) and polymer-film filter (Q). (For I-L and N-Q, UHC = Uncleaned Device Heatstick). (R) Unused and used whole polymer-film filters showing discoloration and film melting, as demonstrated by the narrowing of the used filter. (S) GC-MS Headspace analysis of unused polymer-film filter. Chromatogram shows an overlay of three runs, relative abundance was plotted versus retention time in minutes, unidentifiable peaks were unlabeled. Inset shows a magnified view of peaks with close retention times.