Droplet size measurement of e-cigarette aerosol

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Introduction

• New product category

• Condensation aerosol
  • Physical formation similar to cigarette, but chemistry very different

• Aerosol defined by particle diameter and concentration
  • Limited published data

• Development of robust measurement methods for product assessment, stewardship and potential regulatory assessment
Published data

- Ingebrethsen *et al*, 2012
  - Electrical mobility
  - Multi-wavelength light extinction
- Cartomiser systems
  - Demonstrated importance of square wave profile
  - Demonstrated need for extended sampling times >2s
  - Increased mass output with increased operating time

- For 55 ml puff over 3s
  - Electrical mobility $d_{AM} = 23 - 40$ nm  \# = 2.7 – 7.4e9/cm³
    - Dilution : 3500-5500 : 1
  - Multi $\lambda$ extinction $d_{AM} = 303 - 402$ nm

Methods
Mobility

DMS-500 + Smoking Cycle Simulator (SCS) (Cambustion, UK)

- Reduced pressure so potential for droplet evaporation (0.25 atm)
- 5 – 1000 nm
- 10 Hz resolution
Mobility
DMS-500 + Smoking Cycle Simulator (SCS) (Cambustion, UK)
Laser diffraction

Spraytec (Malvern, UK) + SCS (Cambustion, UK)

- Reduced scatter intensity: \( d_p < \lambda_{vis} \)
- 100 nm – 900 µm
- 2.5 kHz data capture
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Smoking Cycle Simulator

- Variable puff flow 0-100 ml.s\(^{-1}\) at 25 Hz
- Primary dilution into constant total flow up to 8 – 100 l.min\(^{-1}\)
- Secondary dilution available
- Constant Volume Sampling principle for integrated particle number / volume

Operation:
- constant flow 8 l.min\(^{-1}\)
- square wave profile
- 3s duration
- 45, 50, 55, 80 mL volumes
- Max dilution = 9 x (primary only)
Puff by Puff Particle Diameter

3R4F reference cigarette @ 55 / 2 / 30 : 12 puffs
e-cigarette A2 @ 50 / 3 / 30 : Puffs 36 – 48
Cambustion DMS500 and SCS

Note: set of 12 e-cigarette puffs for illustration: RSD = 4.4% over 432 puffs
Puff by Puff Particle Number

3R4F reference cigarette @ 55 / 2 / 30 : 12 puffs (n=5 : 30x secondary dilution)
e-cigarette A @ 50 / 3 / 30 : Puffs 1 - 432 in groups of 12
Cambustion DMS500 and SCS

3R4F cigarette : Increasing particle number with puff (reduced coagulation time + more tobacco mass burnt)
e-cigarette A
Precision: Mobility versus optical diameter

- $VMD_{mob} = 268 \pm 14 \text{ nm (CoV = 5.3\%)}$
  - Cartomiser; rechargeable (UK)
  - 4 devices x 130 puffs

- $VMD_{opt} = 424 \pm 13 \text{ nm (CoV = 3\%)}$
Precision: Mobility diameter

- Cartomiser; rechargeable (UK)
  - 3 devices x 130 puffs
  - VMD = 388 ± 7 nm (CoV = 2%)

- Tank system (CN)
  - 3 devices x 130 puffs
  - VMD = 361 ± 99 nm (CoV = 27%)
Precision: Number concentration (mobility)

- Cartomiser; rechargeable (UK)
  - 3 devices x 130 puffs
  - $N = 7.0 \times 10^9 \pm 7.0 \times 10^8$ (CoV = 10%)

- Tank system (CN)
  - 3 devices x 130 puffs
  - $N = 6.5 \times 10^9 \pm 1.9 \times 10^8$ (CoV = 29%)
Test variables: optical diameter

**MEASUREMENT CAMPAIGN**

Data were reported as median volume-weighted diameters ($d_{v50}$) as follows:

- University of Kentucky 3R4F research cigarette: EM only
- e-cigarette A: EM: $n = 432$ (144 50 ml puffs from each of 3 devices): LD: $n=12$
- e-cigarettes B C D E F: commercial rechargeable products: 55 mL and 80mL puffs
- e-cigarettes B C D: comparison of menthol versus non-menthol variants
- e-cigarettes B-F: $n = 30 : 10$ puffs from each of 3 devices

<table>
<thead>
<tr>
<th>Product</th>
<th>Menthol ±</th>
<th>$d_{v50}$ (nm) 55 ± 30</th>
<th>$d_{v50}$ (nm) 80 ± 30</th>
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</thead>
<tbody>
<tr>
<td>3R4F cig</td>
<td>-</td>
<td>120-180 (EM)</td>
<td>-</td>
</tr>
<tr>
<td>A</td>
<td>-</td>
<td>251 ± 10 (EM)</td>
<td>-</td>
</tr>
<tr>
<td>A</td>
<td>-</td>
<td>430 ± 40 (LD)</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>-</td>
<td>432 ± 22</td>
<td>422 ± 19</td>
</tr>
<tr>
<td>B</td>
<td>+</td>
<td>420 ± 25</td>
<td>425 ± 17</td>
</tr>
<tr>
<td>C</td>
<td>-</td>
<td>432 ± 19</td>
<td>424 ± 11</td>
</tr>
<tr>
<td>C</td>
<td>+</td>
<td>453 ± 21</td>
<td>420 ± 19</td>
</tr>
<tr>
<td>D</td>
<td>-</td>
<td>437 ± 22</td>
<td>415 ± 7</td>
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<tr>
<td>D</td>
<td>+</td>
<td>436 ± 15</td>
<td>422 ± 17</td>
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<tr>
<td>E</td>
<td>-</td>
<td>452 ± 20</td>
<td>430 ± 21</td>
</tr>
<tr>
<td>F</td>
<td>-</td>
<td>443 ± 25</td>
<td>420 ± 11</td>
</tr>
</tbody>
</table>
ANOVA Effects Plot

Difference between products not shown
Regulatory measurement
Data Analysis – Product Specification

- published standards for the validity of Laser Diffraction procedures as a relevant test methodology for medicinal substances\(^1,2\)
- acceptance criteria for \(x_{50}\) data (central distribution value) are specified as \(S_{rel} \leq 10\%\) over at least 6 replicates
- additionally, the acceptance criteria for \(x_{10}\) and \(x_{90}\) data are specified as \(S_{rel} \leq 15\%\) over at least 6 replicates
- criteria must be doubled for values below 10 µm in size

Summary

- Aerosol measurements by electrical mobility and light scattering techniques using a common puffing interface
- Good precision for aerosol diameter; but absolute values differ through evaporation losses in the mobility instrument
- Good precision for aerosol concentration by electrical mobility; requires further validation for optical method
- Development focus on further validation of optical method