How Square is a Square Wave?

A Comparison of Sampling Methods for Electronic Cigarette Emissions and Effect on Air Flow

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Introduction

Current methods published for sampling e-cigarette vapour include CORESTA 81 and the French AFNOR Standard XP D 90-300-3. Both methods state that the square wave of air-flow generated by the "puffing machine" must fit to within set criteria. Aerosol capture equipment is often positioned between the sampling port and the e-cigarette, and as such, may affect the actual air flow applied to the e-cigarette. Also the point at which airflow is measured in relation to the experimental set-up is likely to be critical.

Any variation in the airflow profile between sampling methods may have an effect on the performance of the tested e-cigarette (e.g. evaporative cooling or the amount of air-flow for which a button pushed device is activated). This may in turn incur variation in results between sampling methods and so is important to understand when considering standard methods.

Setup / Method

Setup and Calibration: (Figure 1):

- A Cerulean E-Cigarette Testing Instrument (CETI 8) was used at the following puff profile : 55 mL puff volume, 3 second puff duration, 30 second frequency (55/3/30 in line with AFNOR and CORESTA methods).
- For each of the aerosol capture methods detailed in Table 1, the CETI 8 was set up as follows: Sample Port → Aerosol capture equipment → Flow Meter → E-cigarette. Connections were made with vacuum tubing similar to that of the CETI 8 of differing diameters depending on the connection required (set up diagram in Fig 1, aerosol capture equipment details listed).
- The puff volume was verified using a calibrated oral syringe (immersed in water), and validated for repeatability (n=8).
- The flow meter reading was validated against the verified 55ml puff volume.

Sampling Method:

- For the purposes of this experiment all sampling was performed "dry" (i.e. without activation of the e-cigarette), to avoid the e-cigarette vapour damaging the airflow meter / affecting readings.
- For each setup configuration, 6-8 repeat puff cycles were drawn through the system, and the air flow recorded for each.

Results

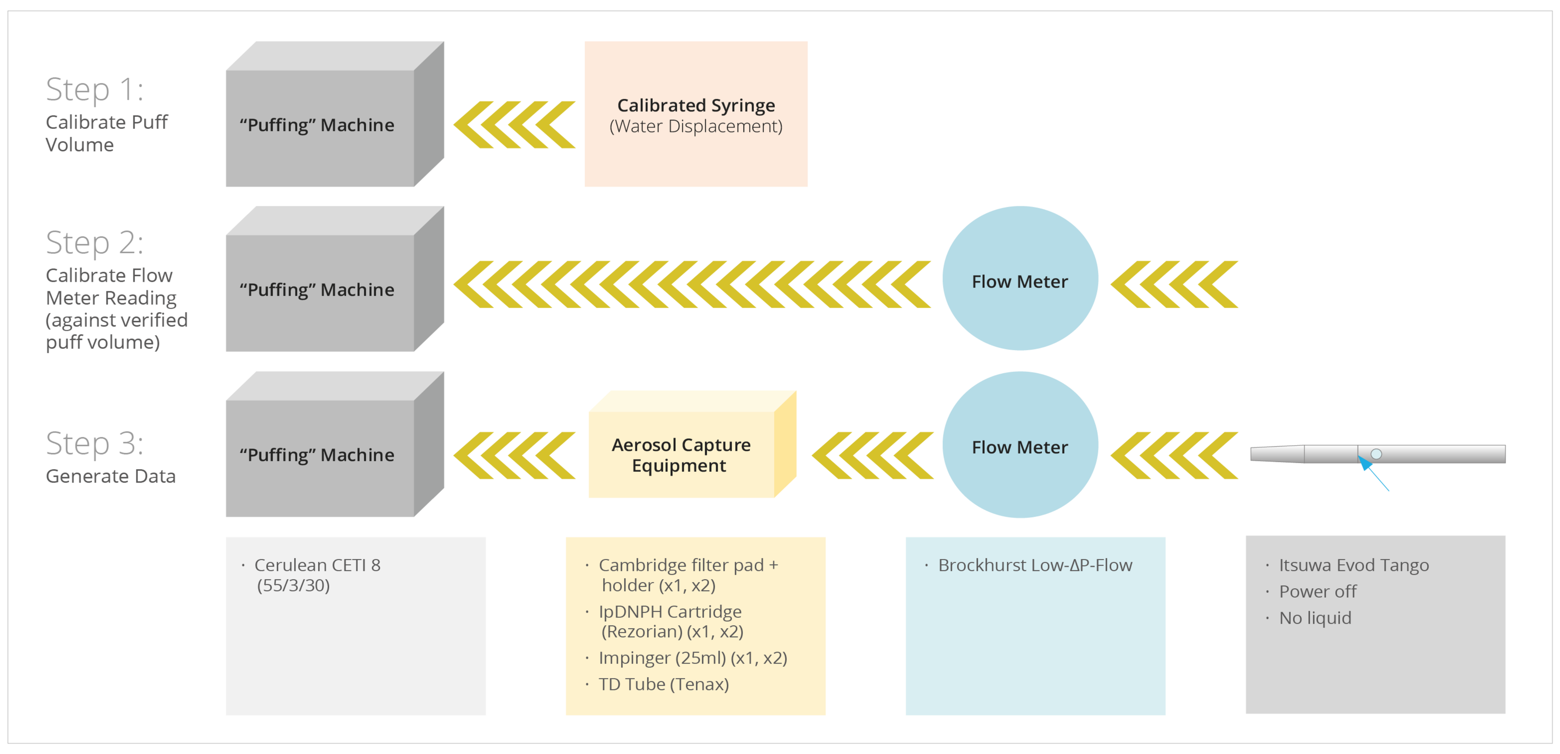
Data Analysis

- Air flow curves were plotted for each data set (n=6-8). Repeat readings within data sets were overlaid, and mean curve plotted. *(Good visual fit was found for all repeats, however as there was a limit in resolution of 0.1 s intervals, analysis may be improved in future studies by use of more sophisticated data manipulation / graphing software).*
- The area under the verified 55 mL puff curve was calculated. By dividing the area under this curve by 55 mL, a calibration factor was calculated to calibrate the remaining sample air flow readings.
- Sample air flow curves were plotted in mL/s against a theoretical 55/3/30 square wave for visual comparison. Puff volumes were calculated (total, within, and outside a theoretical 3 s device activation time).

Results

- The "device only" reading showed a total volume within 1% of the verified reading, there was however a noticeable delay in equilibration, with 11% of the total volume of air drawn after the 3 second activation period, this was similar to the verified 55 mL reading.
- Cambridge filter pads, both single and double showed little change in curve shape compared to the baseline device reading, it should be noted however that these tests were conducted "dry" (i.e. without aerosol), and it is anticipated that resistance may increase as the pads are wetted, and thus the difference will increase.
- Single IpDNPH tubes and 25 mL impingers both showed notable loss in both volume (6%) and maximum air flow (5% and 4% respectively), these values both approximately doubled with the addition of a second sampling item.
- "Maximum air flow" closely correlated with "total volume"

Figure 1: Equipment Setup



**IpDNPH tubes are 2,4-dinitrophenylhydrazine sampling tubes used for carbonyls collection*

Figures 2: Comparison of Airflow Profile Using Each Sample Set-Up

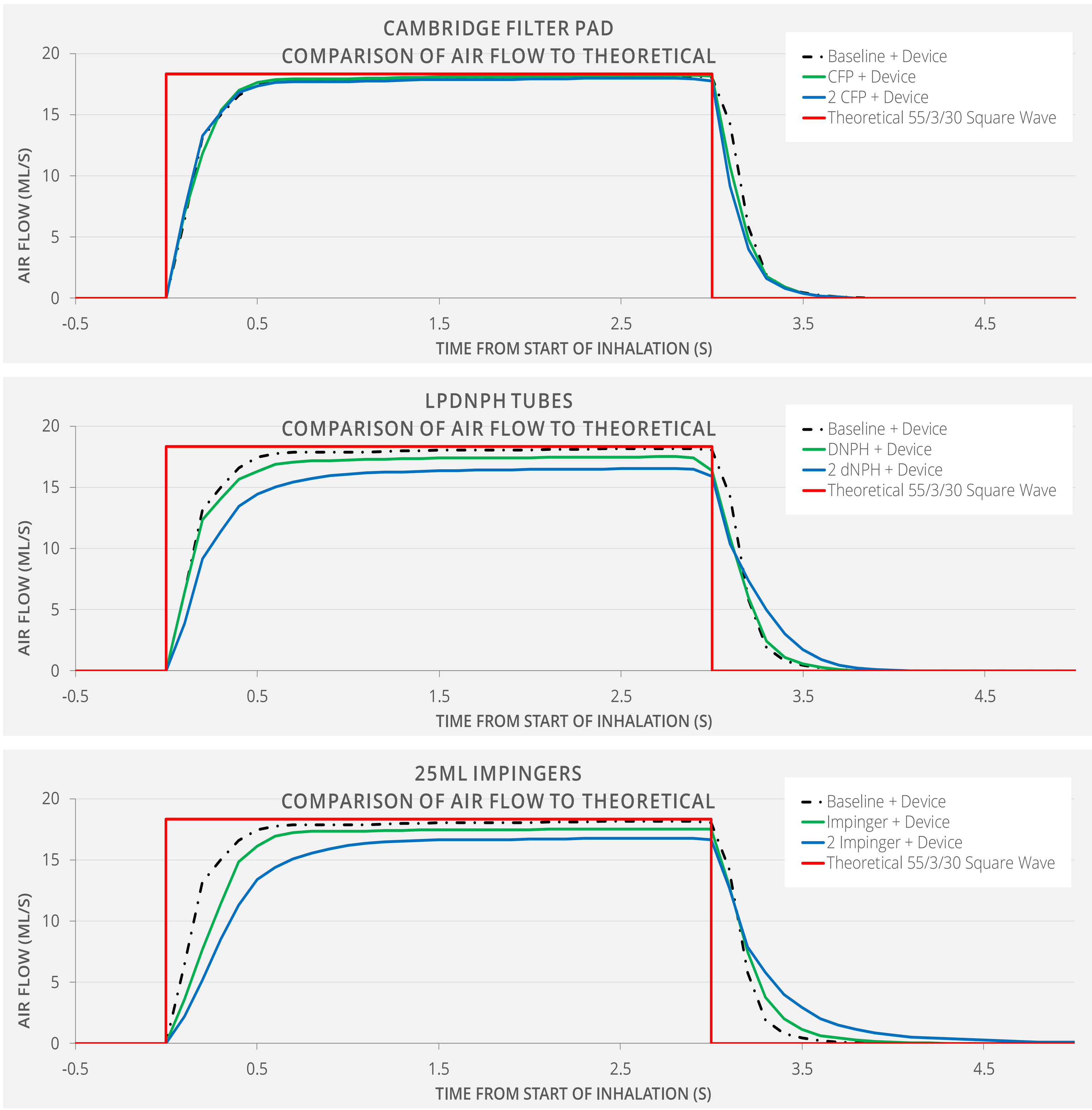


Table 1: Results

Test Setup		Total Volume (mL and % of target)	Volume Outside 3s (mL and % of total)	Volume Inside 3s (mL and % of total)	Max Air Flow (mL/s and % of target)
	Verified 55ml Baseline	55.0 (100%)	5.0 (9%)	50.0 (91%)	18.6 (102%)
	Device Only	54.3 (99%)	5.1 (11%)	49.2 (90%)	18.2 (99%)
Cambridge Filter Pad	single	54.0 (98%)	4.6 (10%)	49.4 (90%)	18.2 (99%)
	double	53.3 (97%)	4.3 (11%)	49.0 (89%)	18.0 (98%)
IpDNPH tubes	single	51.9 (94%)	4.7 (14%)	47.3 (86%)	17.5 (95%)
	double	48.8 (89%)	5.3 (21%)	43.4 (79%)	16.5 (90%)
25mL Impinger	single	51.9 (94%)	5.5 (16%)	46.3 (84%)	17.6 (96%)
	double	49.3 (90%)	6.7 (23%)	42.6 (77%)	16.8 (92%)

Conclusions

Aerosol capture equipment and how it is set up impacts the actual puff profile for the e-cigarette. It is also likely that other aspects of experimental set-up including the e-cigarette will also have an impact. The baseline puff profile was confirmed to be skewed from the theoretical square wave without additional aerosol capture equipment, the shape of the curve was also different to that proposed in CORESTA and AFNOR.

Three main factors were hypothesised to cause distortion of the air flow curve, and as such should be considered for control in any sampling set-up.

- **Resistance of the aerosol capture equipment:** May incur delay in equilibration; increase leakage in the system; and if critical flow is reached, reduce the maximum air flow.
- **"Dead volume" in the system:** May incur delay in equilibration
- **Leakage of the system:** May cause a decrease in the maximum air flow rate.

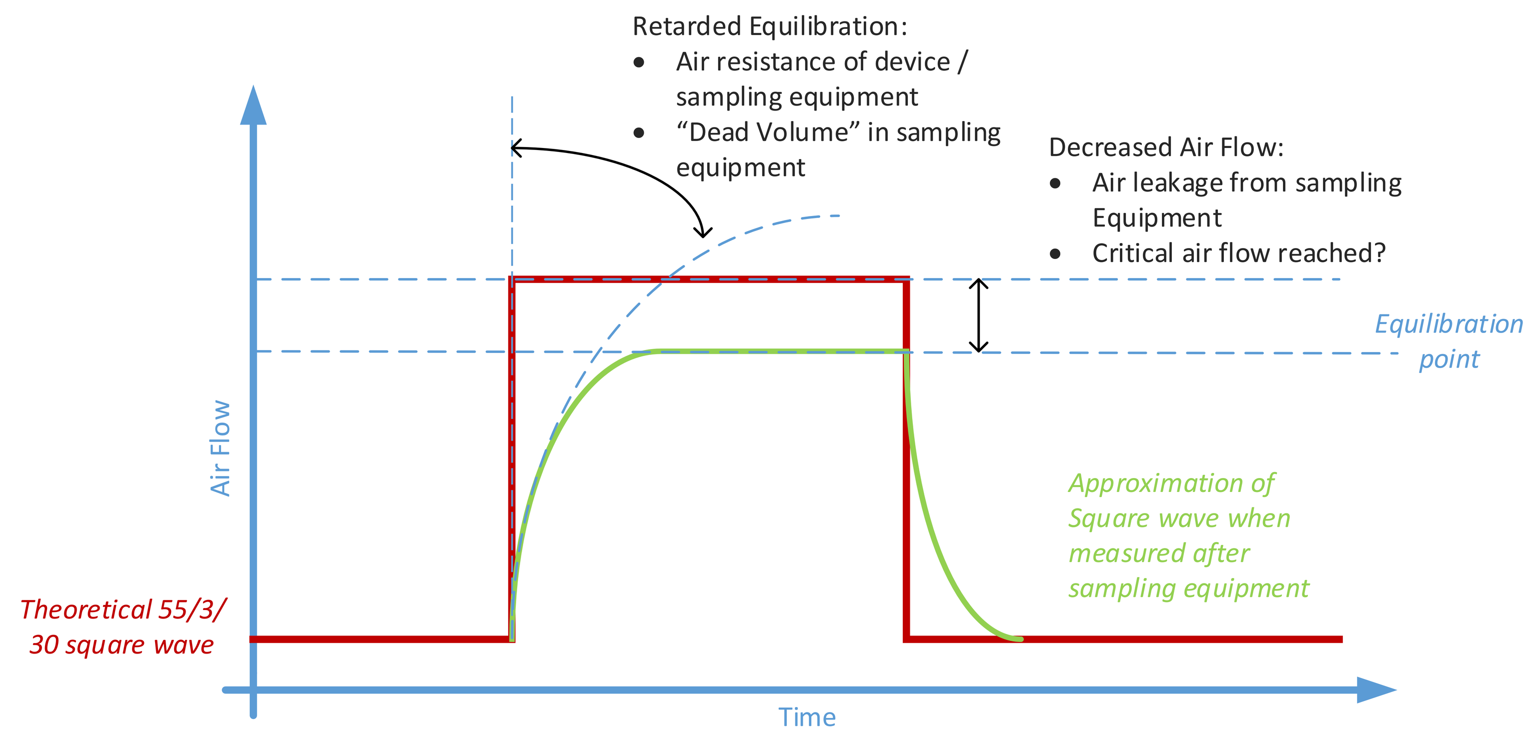
The two main distortions on the airflow profiles, and their possible effect on e-cigarette performance were identified as follows:

- **Reduced maximum air flow:** Shown to reduce the total puff volume, may also reduce the evaporative cooling effect on the coil (likely to cause a higher temperature coil during use)
- **Retardation of equilibration:** Decreases the proportion of the total volume applied within a 3s activation.

Further Study Opportunity

- **Propose and design a system of producing more repeatable air-flow curves across varying aerosol capture equipment** (control air-flow after aerosol capture equipment rather than before?)
- **Further modelling of the effect of system resistance / dead volume / leakage on resulting air-flow** (repeat study using calibrated air resistance standards / measured dead volumes)
- **Reprocessing of data using more advanced data processing / graphing software** and wider range of aerosol capture equipment (e.g. TD tubes)

Figure 3: Typical Airflow profile with Hypothesised Causes for Distortion



1. CORESTA Method No 81: ROUTINE ANALYTICAL MACHINE FOR E-CIGARETTE AEROSOL GENERATION AND COLLECTION – DEFINITIONS AND STANDARD CONDITIONS (June 2015)
2. AFNOR STANDARD XP D 90-300-3: Electronic cigarettes and e‑liquids - Requirements and test methods for emissions