Real-Time Profiling of Aerosol Size and Chemical Composition: Vapor Product at End-of-Liquid

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Background

- Traditional analysis of vapor product aerosol relies on time-consuming offline methods based on capturing aerosol from multiple sequential puffs (~50) onto a medium, followed by extraction and derivatization
- Real-time analysis can eliminate sample alterations that may occur in offline methods, potentially allowing more accurate and time-resolved measurements on puff-by-puff basis
- Proton Transfer Reaction-Mass Spectrometry (PTR-MS) is a soft chemical ionization technique that can simultaneously analyze and quantify a wide range of organic compounds[1]
- Ionization occurs when proton affinity of analyte is higher than that of reagent ion
- PTR-MS is a more sensitive technique compared to offline methods, enabling quantitation of ppt concentrations
- Analyses may be targeted or untargeted
- Electronic Low Pressure Impactor (ELPI+) measures mass median aerodynamic diameter online using electrical current; no need to manually weigh stages after puff collection
- Particle size is predicted to vary as a function of e-liquid formulation, coil temperature, wick material, puff profile, viscosity, etc
- Chemical and physical characterization of aerosol profile can inform product design and potential toxicological risks
- Three different device designs were tested using one puffing profile to assess differences in chemical and physical aerosol profile using real-time analysis methods

Results

Chemical Profiling

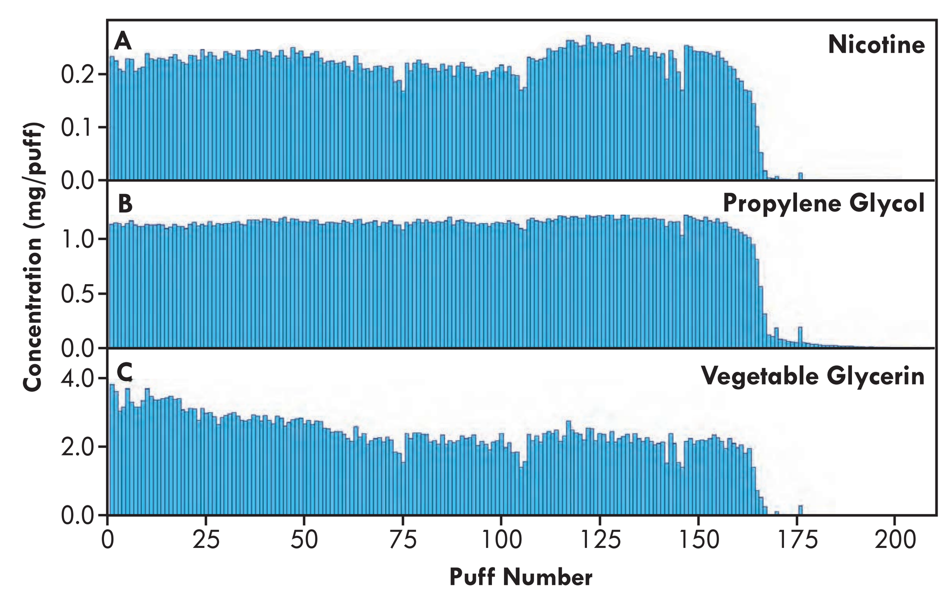

Figure 1. PTR-MS measures individual puffs over the lifetime of a vapor product battery and pod-based cartridge. Analyzing puff-by-puff provides insight into device stability and fluctuations that occur as a function of variables tested, such as liquid remaining in the pod, wicking material, coil temperature, formulation, etc. Shown here are the three main constituents of e-liquids used in vapor products. This technique is easily applied for the analysis of VOCs, and suitable for all chemical compounds with a proton affinity higher than that of H_3O^+.

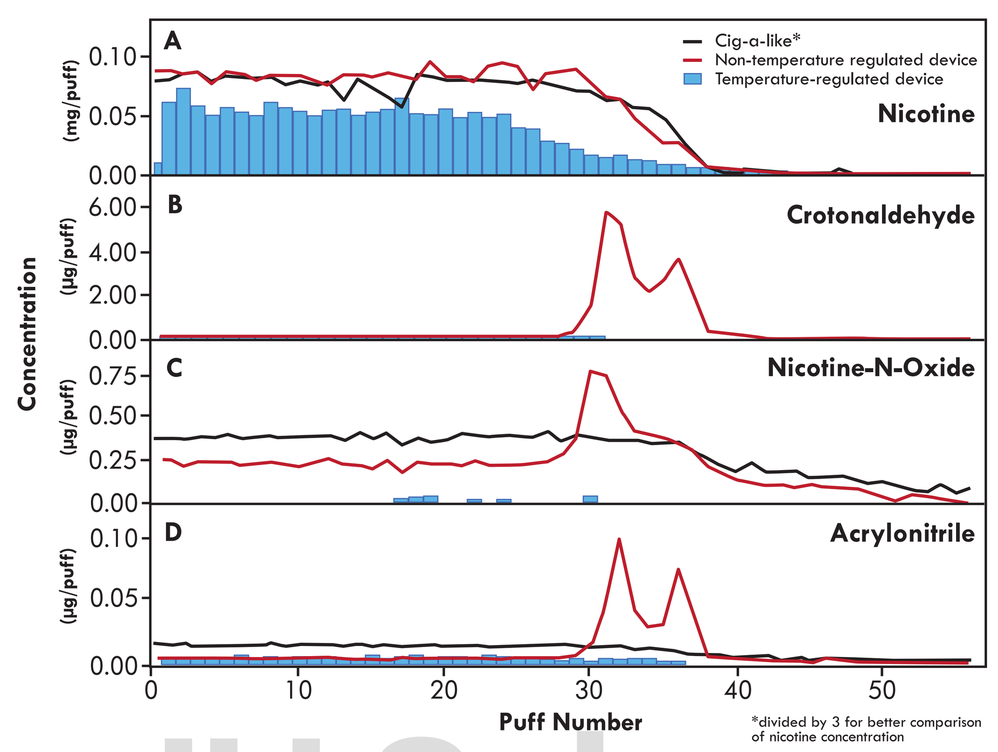

Figure 2. Puff-by-puff analysis of three different device types at end-of-liquid for select analytes (last 40 puffs shown). **A** shows results for nicotine, which predictably drops steadily as the pod liquid runs out. Plots **B**, **C**, and **D** show variances in nicotine degradants and select analytes from FDA's Established List of HPHCs (2012)[2].

Particle Size and Number Profiling

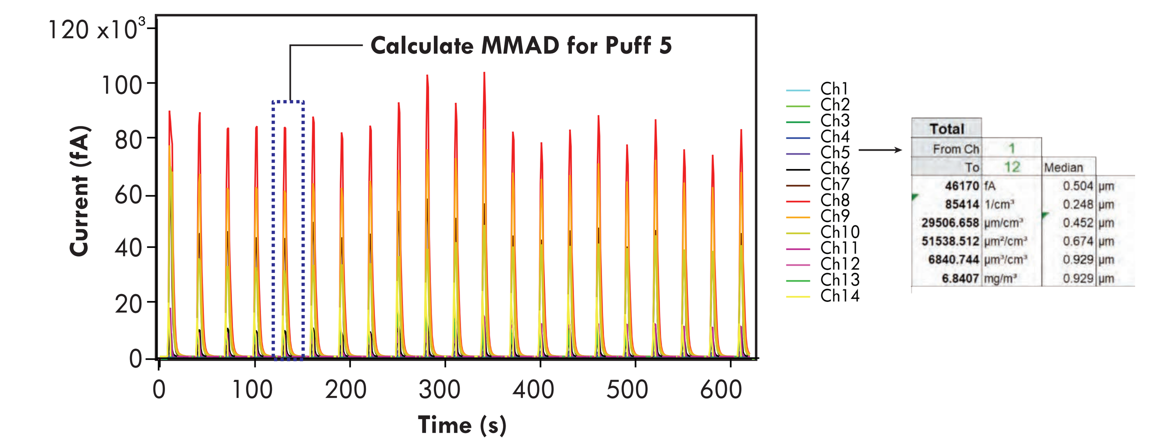

Figure 3. The ELPI+ physical characterization data for all devices was analyzed puff-by-puff. The electrical current measured from each of 14 impactor stages is representative of the number and density of particles in each size range. The measured current is converted to mass median aerodynamic diameter (MMAD) via a proprietary formula. It can also be converted to mass or number concentration.

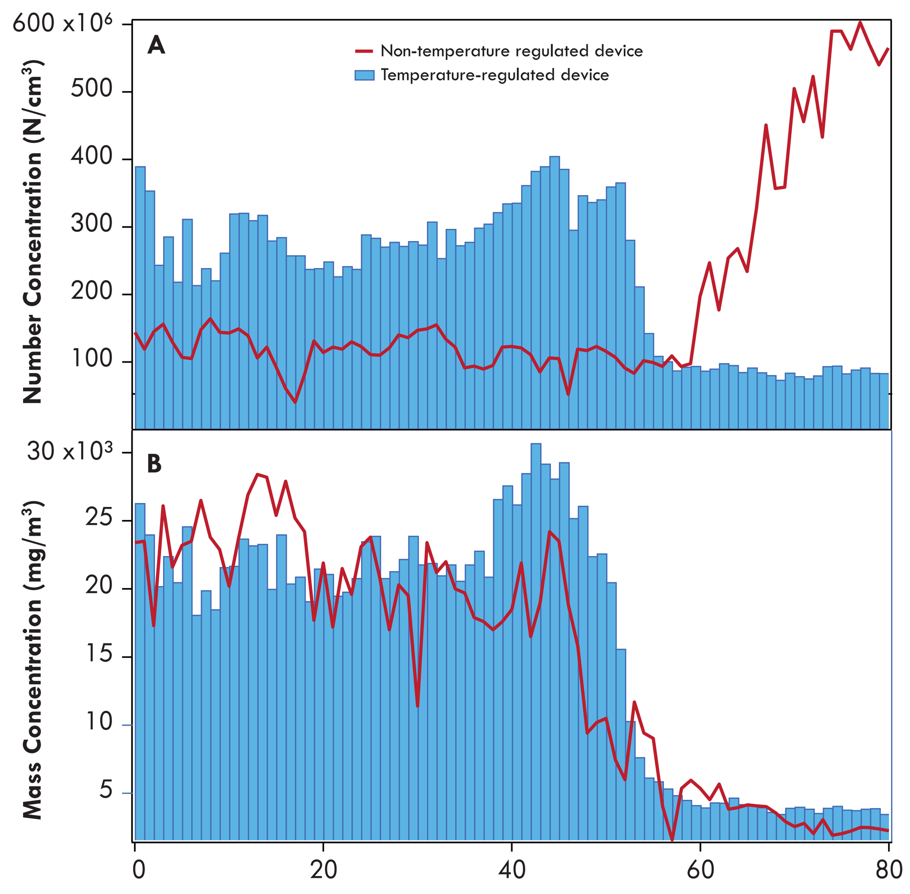

Figure 4. Physical profile comparison at end-of-liquid for **A** temperature-regulated devices, and **B** non-temperature-controlled devices. At end-of-liquid, a spike in number concentration and a corresponding decrease in mass concentration is observed in non-temperature-controlled devices. This, combined with the chemical profile evidence, may suggest that burning occurs as the liquid dries up in non-temperature-controlled devices. For temperature-regulated devices, mass concentration and number concentration both decrease, indicating less aerosol is generated as liquid runs out.

Materials/Methods

Three different device types were analyzed to compare aerosol profiles: a pod-based temperature-regulated device, a pod-based non-temperature-regulated device, and a cig-a-like device. The same puffing profile was used for all devices.

Chemical Profiling

Aerosol is generated by connecting the vapor device to a puffing machine which can be programmed with different puff profiles e.g. 55 mL puff volume for a 3 s puff duration with 30 s between subsequent puffs. The aerosol is vaporized to VOC in heated tubes and subsequently enters a dilutor, where a dilution of 500 up to 1700x takes place. The diluted VOC is then analyzed using the PTR-TOF MS (VOCUS 2R, Tofwerk AG). The analyte is protonated via a proton-transfer reaction with hydronium ions and then separated and detected based on its mass.

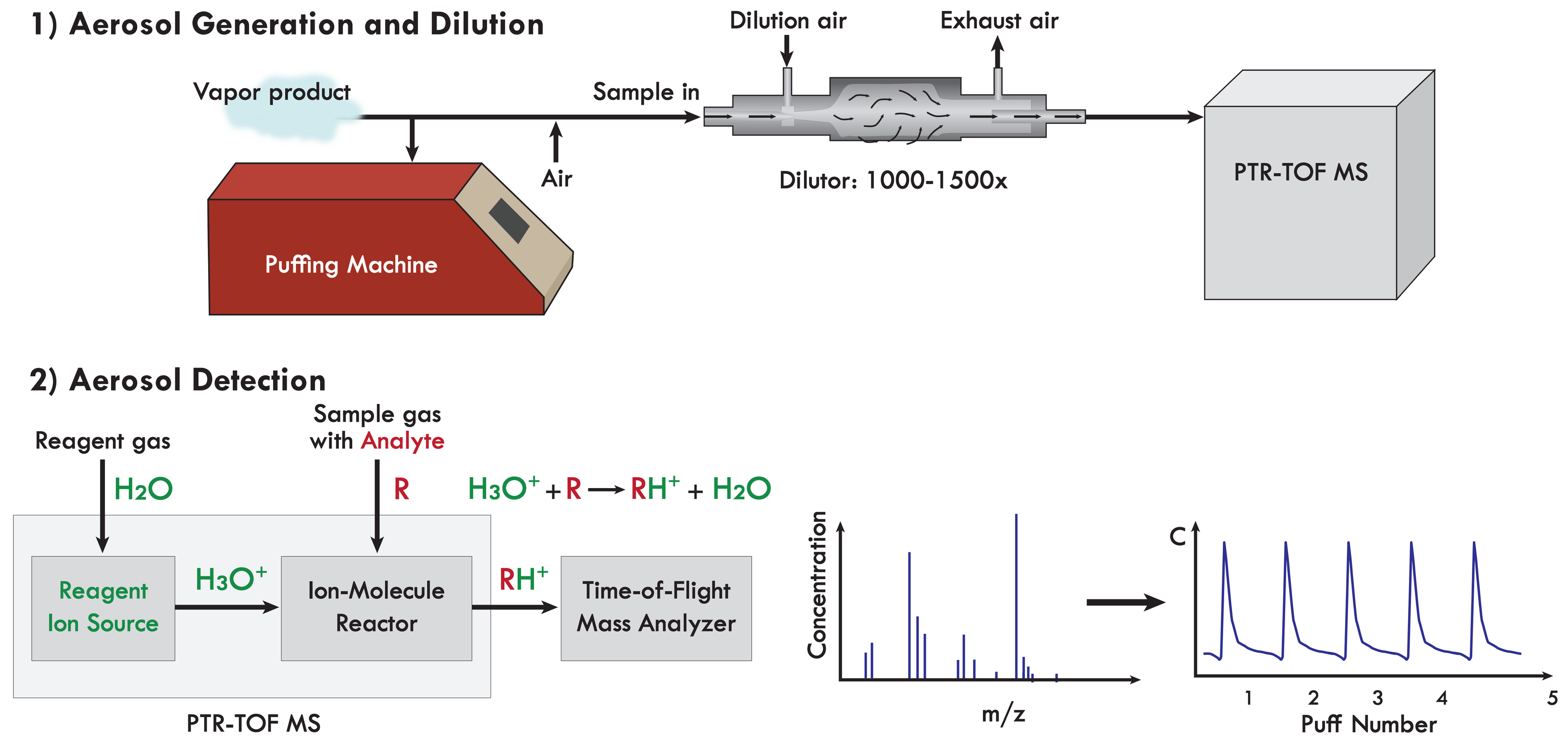

Figure 5. Sampling set-up for chemical profiling of VOCs in aerosol of vapor products using PTR-MS. Aerosol was diluted and vaporized prior to ionization by reagent gas and time-of-flight detection. (Vocus, Tofwerk AG)

Particle Size and Number Profiling

Aerosol is generated by connecting the vapor device to a puffing machine which can be programmed with different puff profiles e.g. 55 mL puff volume for a 3 s puff duration with 30 s between subsequent puffs. The aerosol is diluted approximately 750x with clean dry air before entering the ELPI+. The diluted aerosol is positively charged via an electric field using a corona charger. The charged aerosol enters the impactor where it is separated according to size and measured by electric current.

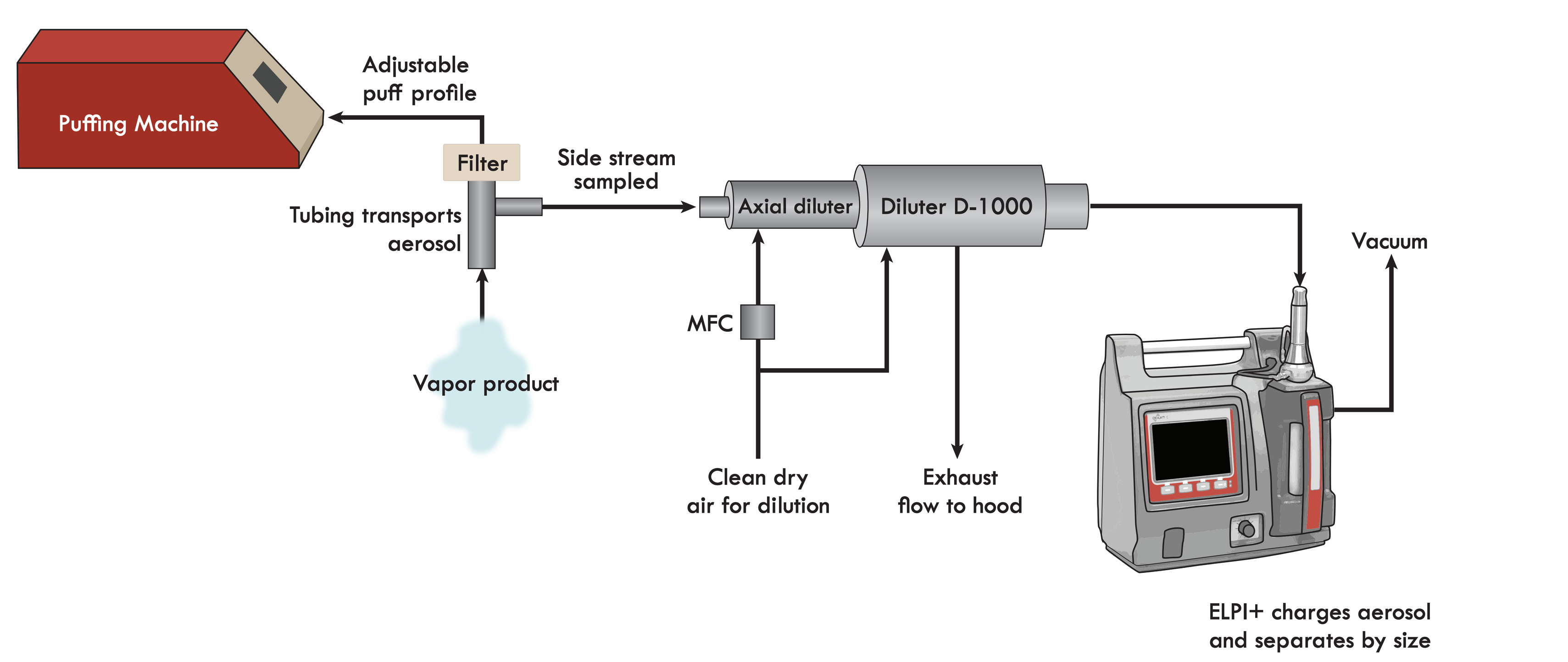

Figure 6. ELPI+ (Dekati) data acquisition schematic for physical characterization. Aerosol from vapor device is sampled and diluted approximately 740:1 before being charged and measured by the instrument. Current (fA) measurements are converted to number and mass distribution via a mathematical formula.

Conclusions

- Online aerosol analysis of HPHCs with puff-to-puff resolution is possible with PTR-MS; puff-by-puff analysis provides necessary insight into device stability and end-of-liquid characterization
- End-of-liquid chemical and physical characterization both show changes in aerosol profile of non-temperature-controlled devices indicative of burning (dry puffing)
- ELPI+ is a viable alternative to offline particle sizing methods that enables puff-by-puff resolution
- Differences in device design result in differences in the chemical-physical profile that support different and deeper investigations related to emissions and exposure assessment

References

1. Breiev, K.; Burseg, K. M. M.; O'Connell, G.; Hartungen, E.; Biel, S. S.; Cahours, X.; Colard, S.; Märk, T. D.; Sulzer, P. An Online Method for the Analysis of Volatile Organic Compounds in Electronic Cigarette Aerosol Based on Proton Transfer Reaction Mass Spectrometry. Rapid Commun. Mass Spectrom. 2016, 30 (6), 691–697. https://doi.org/10.1002/rcm.7487.

2. FDA, Harmful and Potentially Harmful Constituents in Tobacco Products and Tobacco Smoke, Established List (77 Fed. Reg. 20034) (2012)

Note: Consistent with applicable laws and regulations, JUUL Labs does not, and cannot, promote its products as being less harmful than cigarettes.