Gas Phase Infrared Spectroscopy Enables Puff-by-Puff Profiling of a Novel Heated Tobacco Capsule (HTC) Prototype

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Altria

FAMILY OF COMPANIES

Enhanced Understanding of HTP Aerosols Puff-by-Puff Resolution of Nicotine, CO & Formaldehyde

Establish methodology for Puff-by-Puff (P-b-P) characterization of HTP Aerosols

- Gas Phase FTIR

Directly analyze nicotine, CO and formaldehyde in a novel Heated Tobacco Capsule (HTC) prototype

- Simultaneous Measurement

Determine the impact of heating profile changes on analyte levels by puff

- Constituent delivery trends across product use sessions

Compare the findings relative to other HTP products

- Heated tobacco sticks (HTS)

CO=carbon monoxide; FTIR=Fourier Transform Infrared; P-b-P=Puff-by-Puff; HTC=heated tobacco capsule; HTP=heated tobacco product; HTS=heated tobacco sticks.

FTIR Calibration

Chemometric calibrations – multiple analytes | Calibrations (ppm) → mg/unit mg/puff

Development:

- Gas standards
- Nitrogen gas dilution

To improve accuracy of FTIR quantitation:

- Use GC-MS and chromatography data for calibration

FTIR=Fourier Transform Infrared.

Project Overview

2 DEVICES | **6** REPS EACH

Non-menthol (NM) and Menthol (M) variants for each

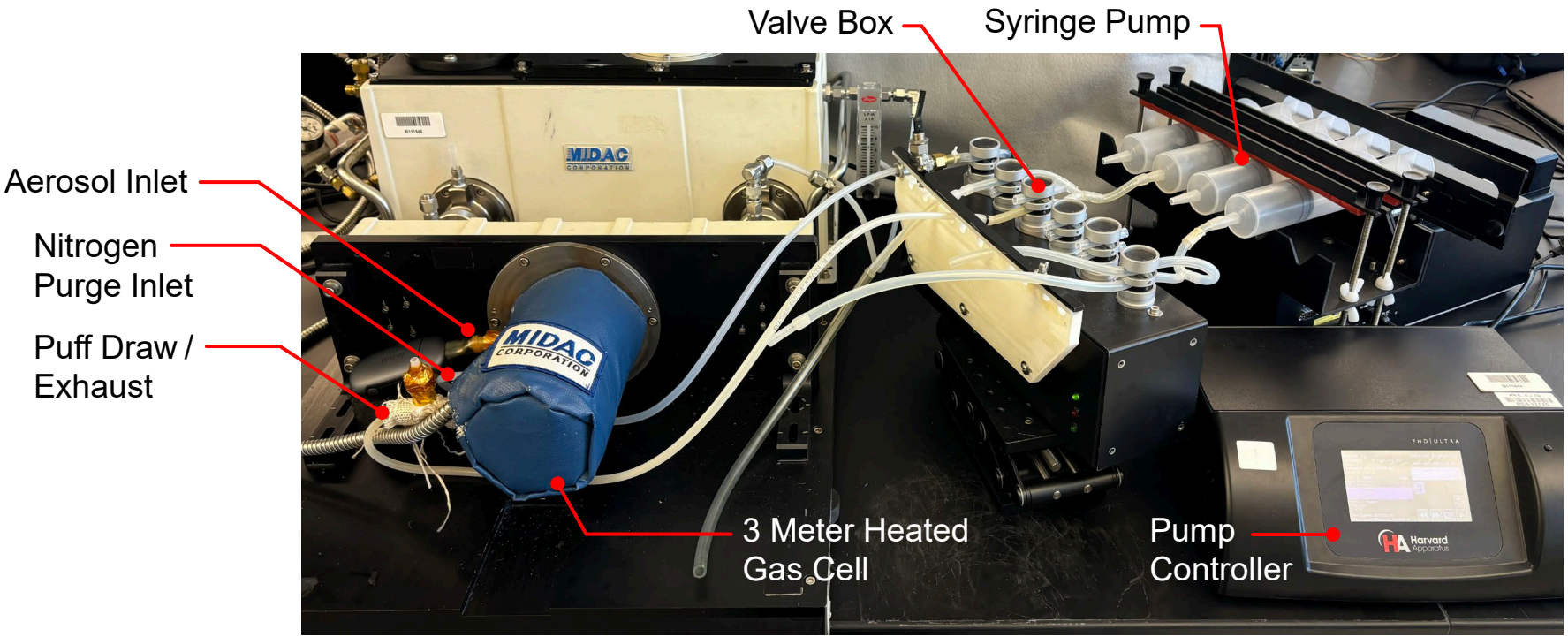
HTC	**14 puffs, 55-2-30**

Heating Profile: Normal | -30°C | +30°C

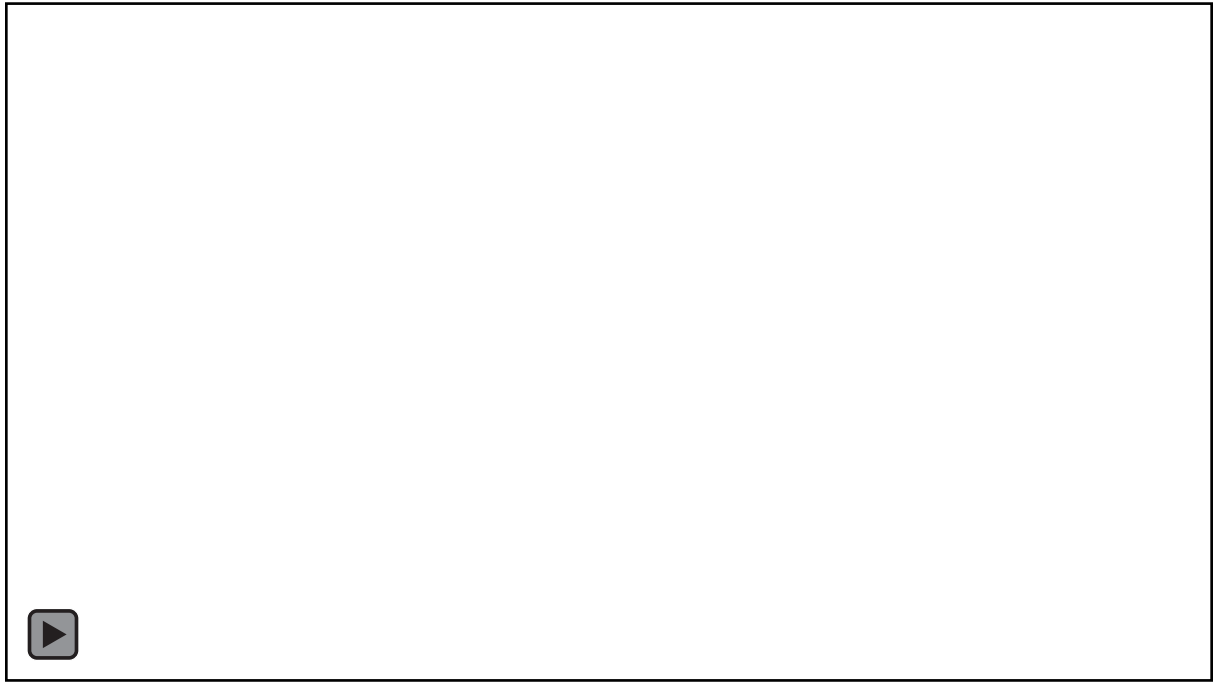
HTP Comp1	**12 puffs, 55-2-30**
HTP Comp2	**8 puffs, 55-2-30**

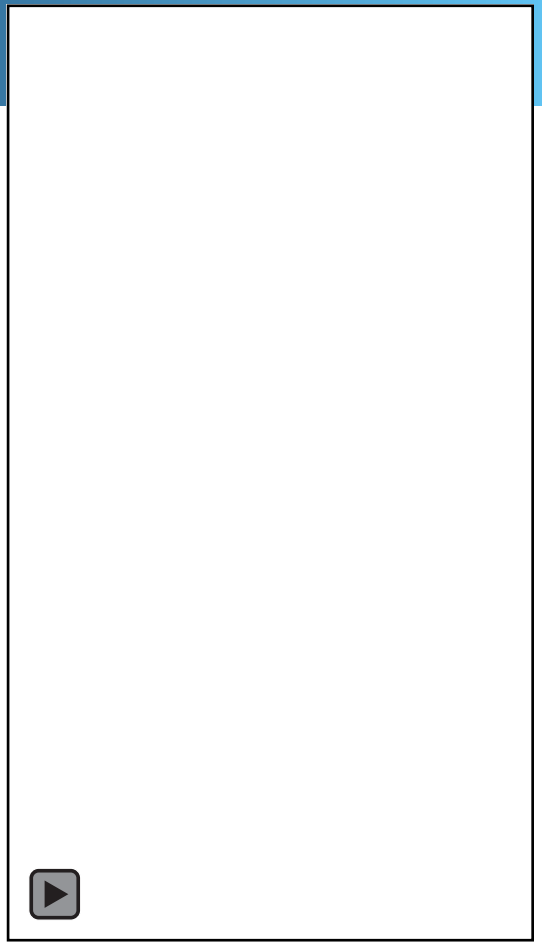
HTC= heated tobacco capsule; HTP=heated tobacco product; Comp=comparator.

FTIR Puff-by-Puff Apparatus



Controlled P-b-P Events



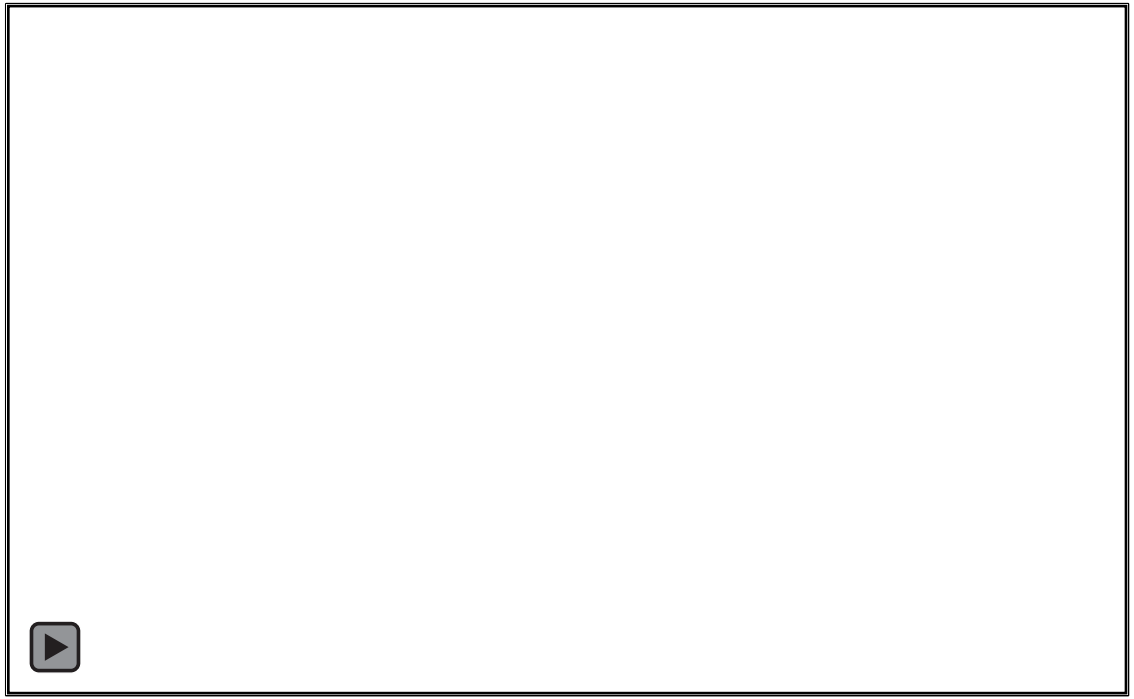


P-b-P=puff-by-puff.

Live FTIR Puff Sequence

Fast & effective aerosol exchange:

- Atmospheric water vapor and CO_2 are shown
- P-b-P steps
  - Intake
  - Hold (scan time)
  - Exhaust



FTIR=Fourier Transform Infrared; CO_2=carbon dioxide; P-b-P=Puff-by-Puff.

HTP Temperature Profiles

How do HTP heater temperature changes effect nicotine, CO and formaldehyde aerosol concentrations on a puff-by-puff basis?

> The literature shows consistency in that below a characteristic temperature of 400°C, thermal degradation of the tobacco in tobacco products is net endothermic, the formation of products that can be related to oxidation is limited, yields of polycyclic aromatic hydrocarbons (PAHs) are negligible and atmospheric oxygen has very limited influence on the degradation chemistry.[1]

Nicotine BP	247°C
HTC Heating Profile Temperature Changes	-30°C Normal +30°C
HTP comparator temperature profiles	**COMP1:** 300–350°C **COMP2:** 200–250°C

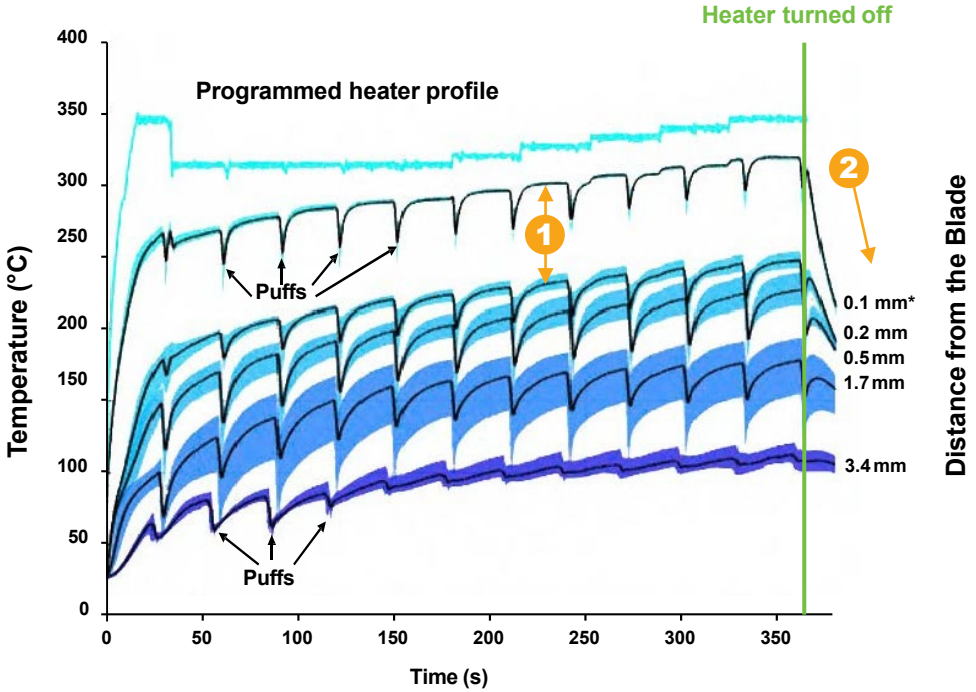
BP=boiling point; CO=carbon monoxide; Comp=comparator; HTC=heated tobacco capsule.

1. Jose Torero Cullen, Teemu Karkela, Unto Tapper. Signatures that differentiate thermal degradation and heterogeneous combustion of tobacco products and their respective emissions; *J. Anal. Appl. Pyrolysis*, 179 (2024) 106478.

Example Heating Profile

Small proportion of tobacco substrate is exposed to higher temps:

1. Nearly 50°C decrease from 0.1mm to 0.2mm
2. Rapid decrease in temperature after the heater is turned off
   - No combustion

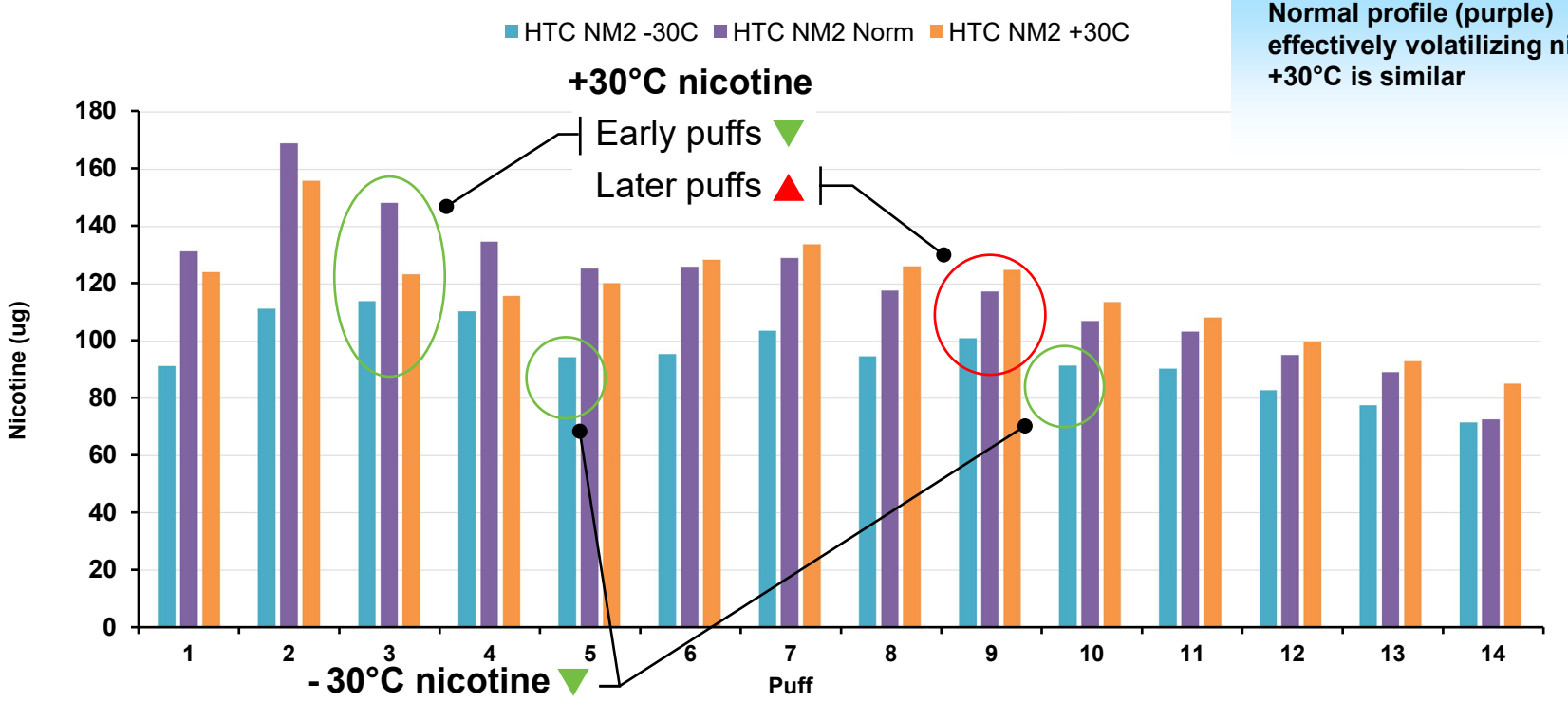


*Radial position of thermocouple relative to the surface of the heater.
The IQOS Heating System, (TPSAC / FDA, Jan 24, 2018). https://www.fda.gov/files/advisory%20committees/published/January-24-25-TPSAC-Meeting-Philip-Morris-International-Back-up-Slides.pdf

Heating Profile Effects – Temp +/- 30°C

Nicotine – P-b-P
HTC NM2

Normal profile (purple) effectively volatilizing nicotine - +30°C is similar

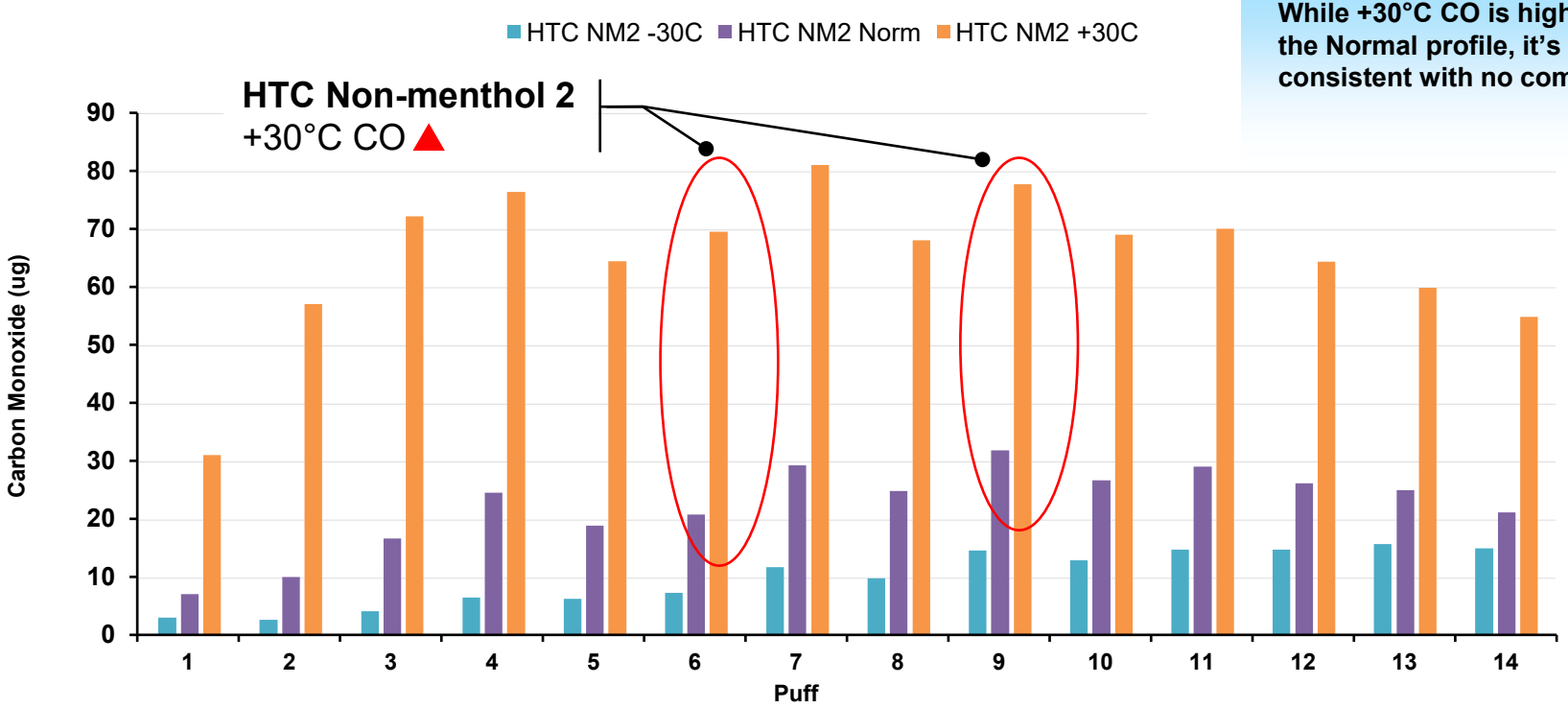


HTC=heated tobacco capsule; NM=non-menthol; Norm=normal; P-b-P=puff-by-puff.

Heating Profile Effects – Temp +/- 30°C

Carbon Monoxide – P-b-P
HTC NM2

While +30°C CO is higher than the Normal profile, it's still consistent with no combustion



HTC=heated tobacco capsule; NM=non-menthol; Norm=normal; P-b-P=puff-by-puff.

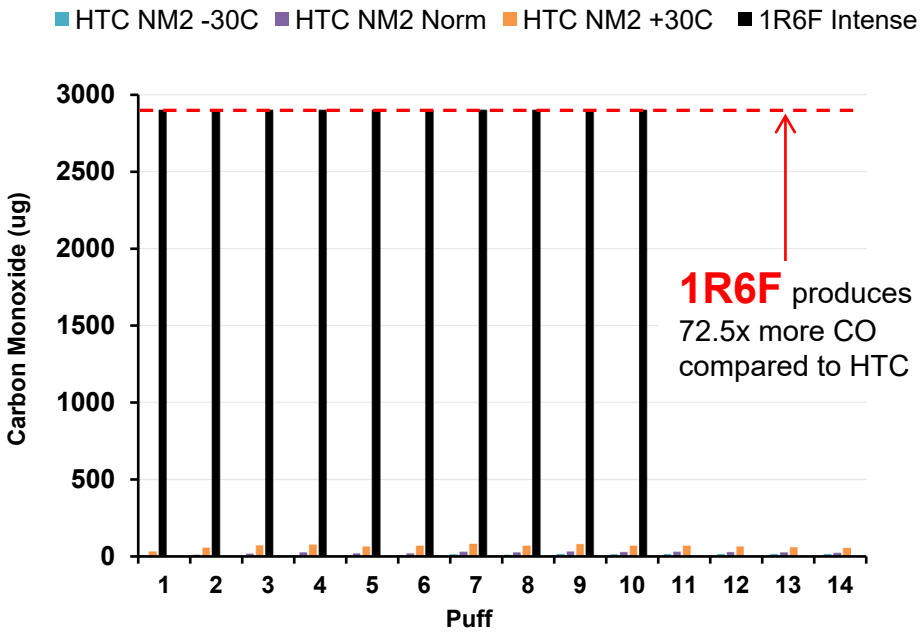
Heating Profile Effects – Temp +/- 30°C

Carbon Monoxide – P-b-P
HTC NM2

Tobacco thermochemical degradation

> "…carbon monoxide is not formed as a combustion product until **460°C**."[1]
>
> "The EHTP tobacco undergoes drying and evaporation during EHTS use, and **only a small portion of the tobacco close to the Heater undergoes thermochemical decomposition** (torrefaction/low-temperature pyrolysis), but no combustion (neither incomplete nor complete) occurs.[2]"

Emphasis added



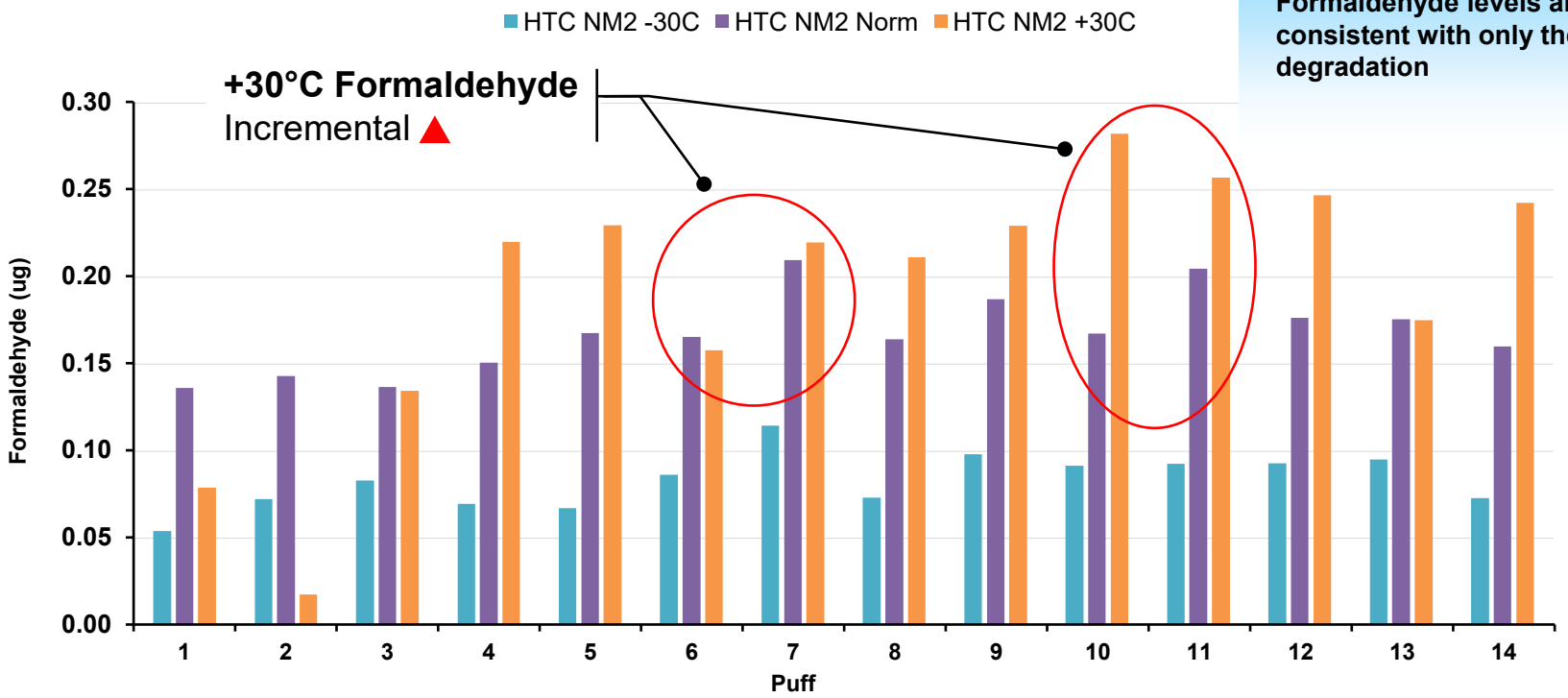
1R6F=1R6F reference cigarette; EHTP=electrically heated tobacco product; EHTS=electrically heated tobacco sticks; HTC=heated tobacco capsule; NM=non-menthol; Norm=normal; P-b-P=puff-by-puff.

1. R.R. Baker. Formation of carbon oxides during tobacco combustion: Pyrolysis studies in the presence of isotopic gases to elucidate reaction sequence; J. Anal. Appl. Pyrolysis, 4 (1983), pp. 297-334.
2. Nordlund M, Smith M, Maeder, S McGrath T, Schaller JP, Pratte P, Picavet P, Peitsch M. Scientific substantiation of the absence of combustion and no smoke formation in the Electrically Heated Tobacco Product (EHTP), version 1.0 [Internet]. Neuchâtel, Switzerland: Philip Morris Products S.A.; 2019 January 21 p. 1-4.

Heating Profile Effects – Temp +/- 30°C

Formaldehyde – P-b-P
HTC NM2

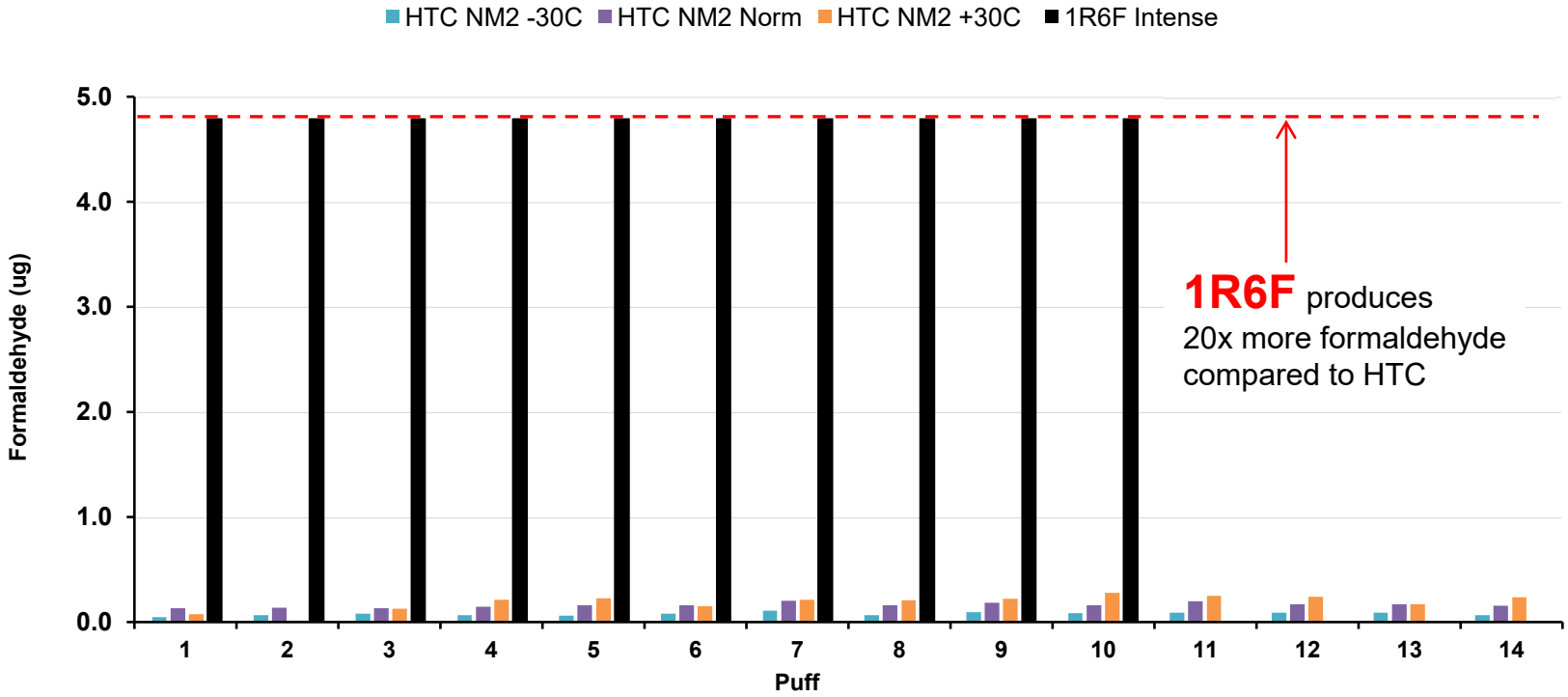
Formaldehyde levels are low – consistent with only thermal degradation



HTC=heated tobacco capsule; NM=non-menthol; Norm=normal; P-b-P=puff-by-puff.

Heating Profile Effects – Temp +/- 30°C

Formaldehyde – P-b-P
HTC NM2



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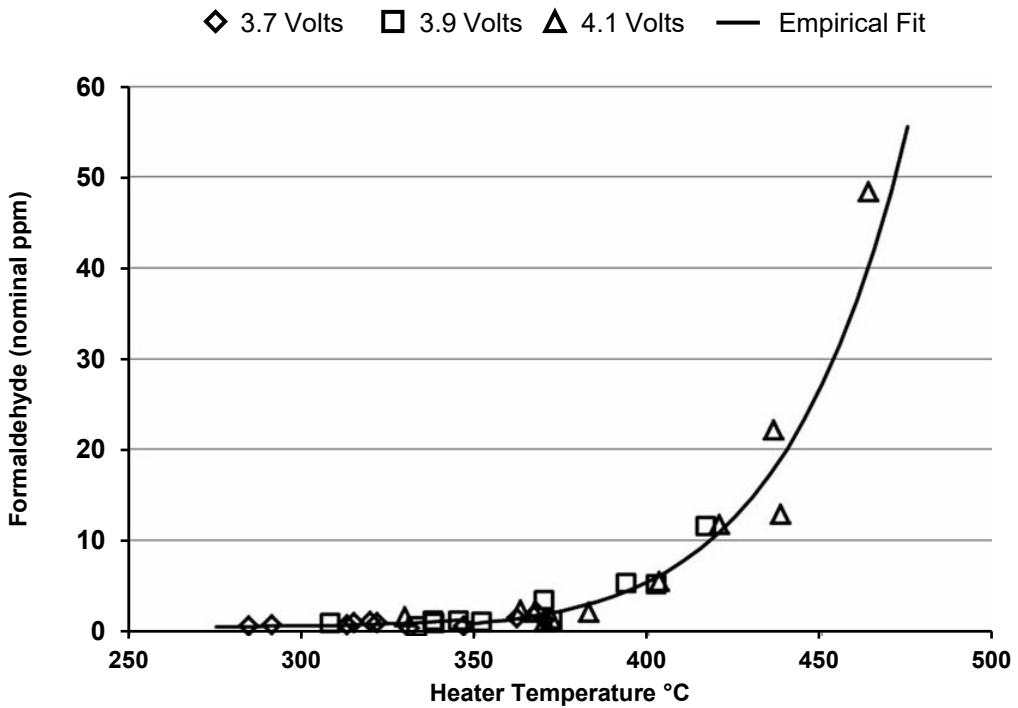
Formaldehyde vs. Heater Temp

Formaldehyde
Very low <350°C,
increases rapidly at 400°C[1]



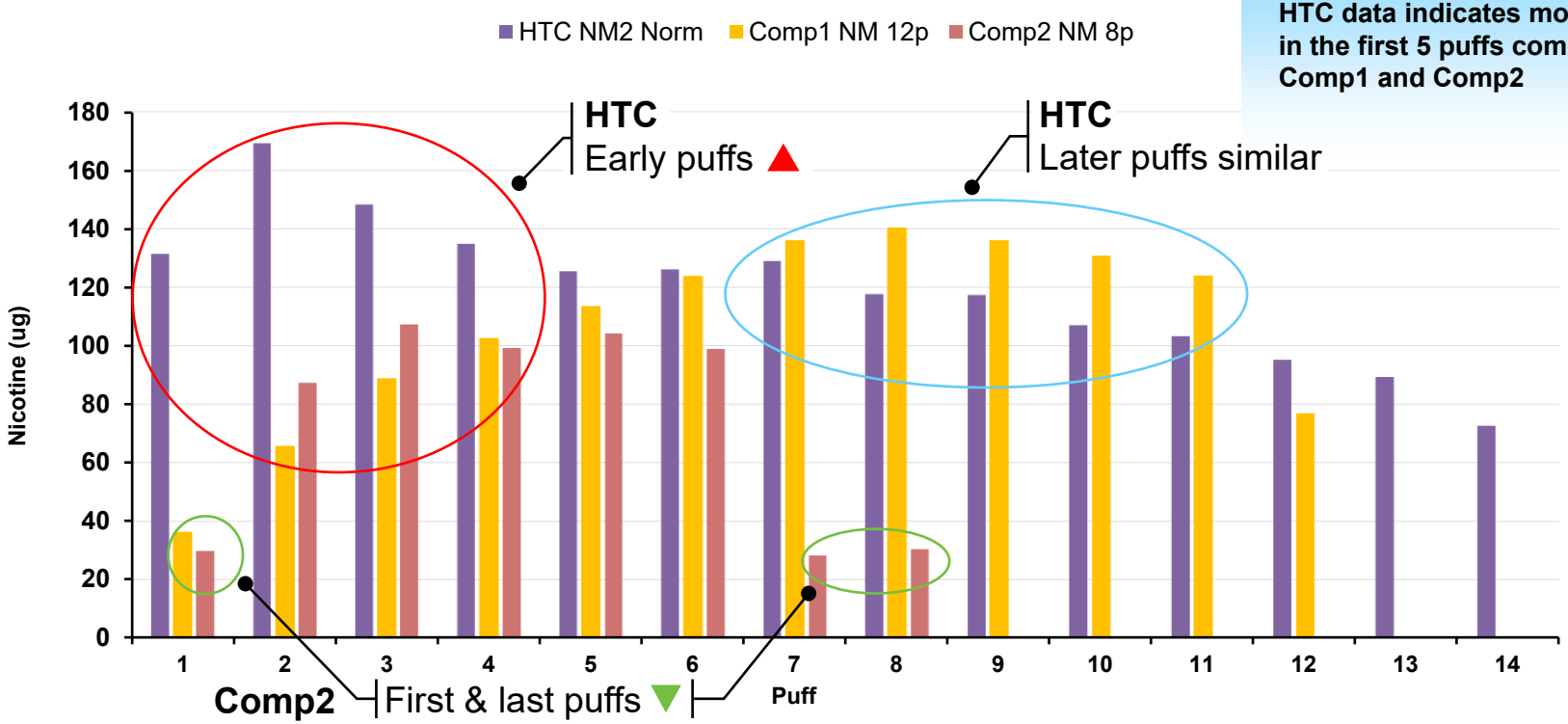
ppm=parts per million.

1. Jason W. Flora*, Celeste T. Wilkinson, James W. Wilkinson, Peter J. Lipowicz, James A. Skapars, Adam Anderson, and John H. Miller. Method for the Determination of Carbonyl Compounds in E-Cigarette Aerosols; *Journal of Chromatographic Science*, (2016), 1–7.

Comparator Results

Nicotine – P-b-P
HTC, Comp1, & Comp2

HTC data indicates more nicotine in the first 5 puffs compared to Comp1 and Comp2

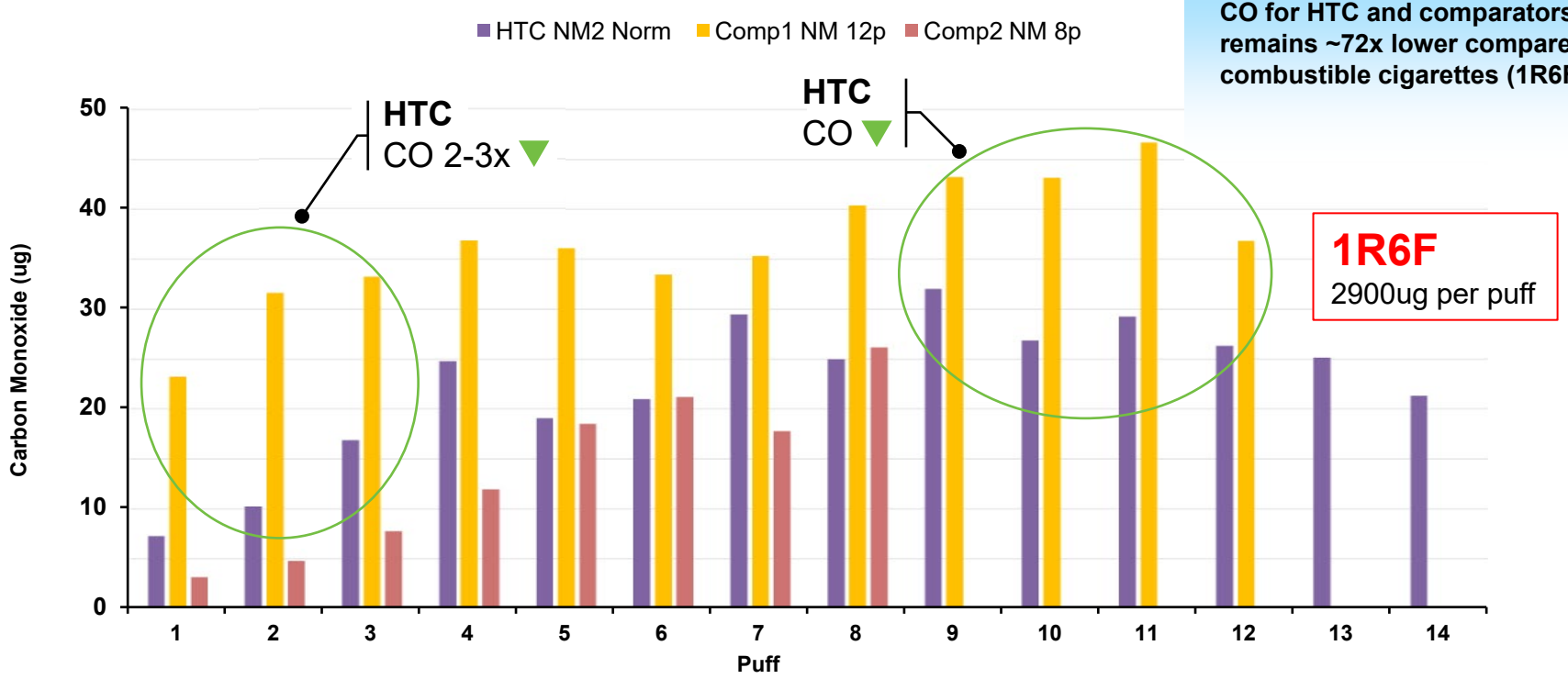


Comp=comparator; HTC=heated tobacco capsule; NM=non-menthol; Norm=normal; P-b-P=puff-by-puff.

Comparator Results

Carbon Monoxide – P-b-P
HTC, Comp1, & Comp2

CO for HTC and comparators remains ~72x lower compared to combustible cigarettes (1R6F)

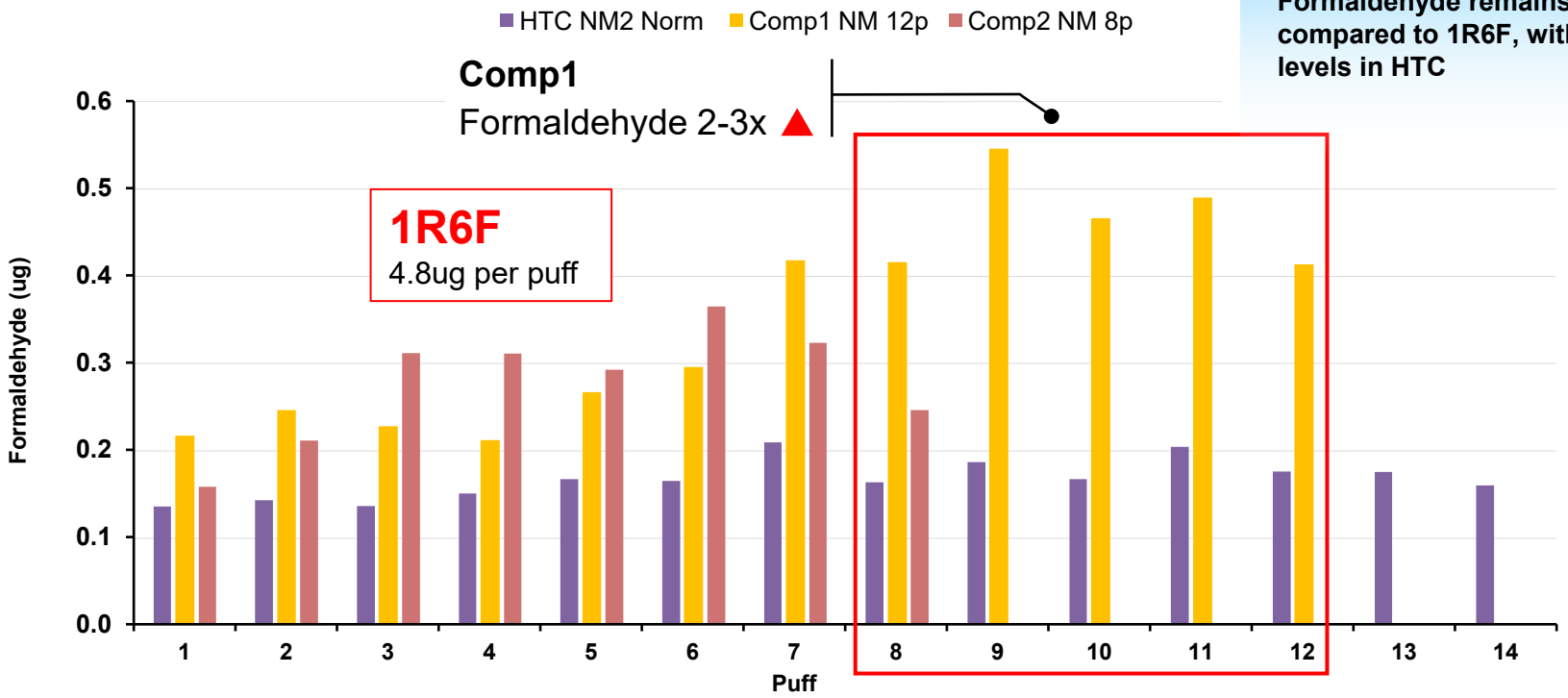


1R6F=1R6F reference cigarette; Comp=comparator; HTC=heated tobacco capsule; NM=non-menthol; Norm=normal; P-b-P=puff-by-puff.

Comparator Results

Formaldehyde – P-b-P
HTC, Comp1, & Comp2

Formaldehyde remains 20x lower compared to 1R6F, with lowest levels in HTC



1R6F=1R6F reference cigarette; Comp=comparator; HTC=heated tobacco capsule; NM=non-menthol; Norm=normal; P-b-P=puff-by-puff.

Summary

FTIR Puff-by-Puff Method

Direct, Real time and simultaneous measurement of nicotine, CO and formaldehyde

- Flexible – menthol or non-menthol, other device types
- Chemometric calibrations

Heating Profile Experiments

- Supports literature statements
  - P-b-p profile changes at endothermic levels
  - CO and formaldehyde are orders of magnitude lower than combustible cigarettes 1R6F
- +30°C no increase in nicotine
  - Incremental increase in CO and formaldehyde
  - Nicotine shift from early to later puffs
- -30°C decrease in nicotine

Comparator Experiments

- Nicotine p-b-p profiles
  - HTC ▲ – first 5 puffs
- CO and Formaldehyde levels consistent with endothermic thermal degradation – very low compared to 1R6F

1R6F=1R6F reference cigarette; CO=carbon monoxide; FTIR=Fourier Transform Infrared; P-b-P=Puff by Puff; HTC=heated tobacco capsule.

Thank you!

Any Questions?

References

1. Jose Torero Cullen, Teemu Karkela, Unto Tapper. Signatures that differentiate thermal degradation and heterogeneous combustion of tobacco products and their respective emissions; J. Anal. Appl. Pyrolysis, 179 (2024) 106478
2. The IQOS Heating System, (TPSAC / FDA, Jan 24, 2018). https://www.fda.gov/files/advisory%20committees/published/January-24-25-TPSAC-Meeting-Philip-Morris-International-Back-up-Slides.pdf
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