

Determination of Aerosol Mass and Five Primary Constituents in Aerosols generated from Heated Tobacco Products

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Abstract

In 2021, CORESTA conducted a proficiency study for the determination of aerosol mass, propylene glycol, glycerin, and nicotine in aerosols generated from heated tobacco products (HTPs) to identify and recommend a suitable method to measure these components. Due to most laboratories using a similar methodology based on ISO 24199 (CRM 84) and ISO 20778 (intense puffing regime), the study was considered to be a proficiency and collaborative study. However, the study did not include the determination of two additional primary HTP aerosol constituents, water, and menthol. Our efforts focused on expanding the method scope to include these two additional constituents. The sample trap assembly was modified to include a Cambridge filter pad (CFP) followed by an impinger containing a solvent to capture the breakthrough of water and menthol, which ranged from 15% to 30%. The modified method was validated using a novel heated tobacco capsule (HTC) prototype, which consists of a hand-held battery-operated device and a disposable tobacco-containing capsule that is inserted into the device. Method modifications and validation results for the determination of Propylene Glycol (PG), Glycerin, Nicotine, Water, and Menthol from the HTC prototypes will be presented. The mean sample recoveries for all analytes ranged from 86.2% to 103.0%. The %RSD of repeatability for each day was $\leq 14.6\%$ for all samples. The %RSD of intermediate precision over 3 days was $\leq 9.0\%$. These method validation elements all met their pre-determined acceptance criteria. This validation data demonstrates that the modified method is suitable and reliable for measuring aerosol mass and the five primary constituents in aerosols generated by HTPs.

Objective

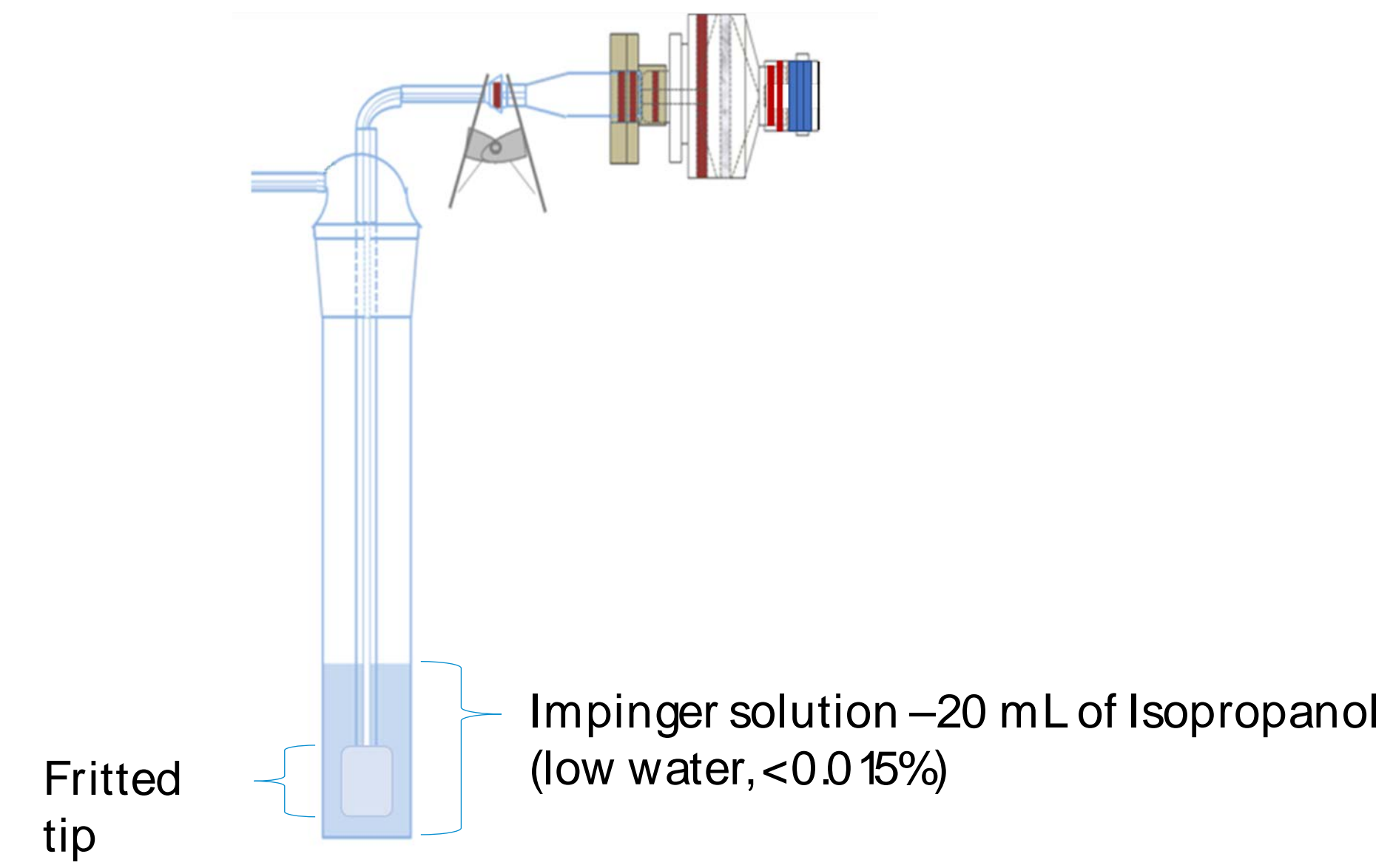
- Develop and validate a method that is fit for purpose to measure Propylene Glycol, Glycerin, Nicotine, Menthol, and Water in aerosol emissions generated from Heated Tobacco Products (HTPs) using a novel heated tobacco capsule (HTC) prototype and comparator HTPs that use heated tobacco sticks (HTS).
- Utilize the validated method to compare analytes in aerosols of HTC prototypes against those in aerosols of HTPs that use HTS.

Method Principle

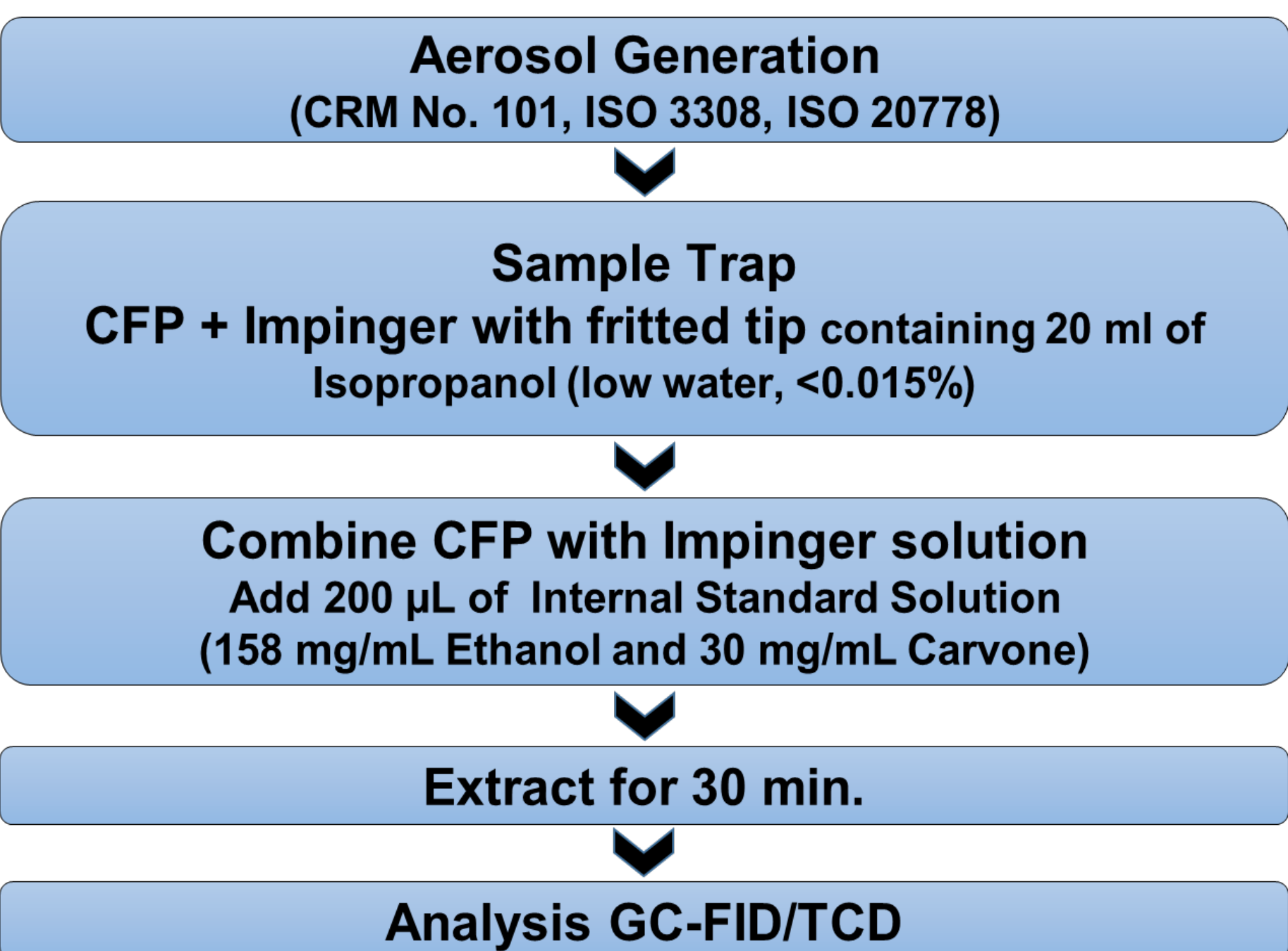
Aerosol emissions from HTPs are generated and collected using ISO 3308 (non-intense) and ISO 20778 (Intense) puffing regimes and procedures described in CRM No. 101. The sample trap for HTP aerosol emissions includes a Cambridge filter pad followed by an impinger with a fritted tip containing 20 mL Isopropanol. Aerosol Mass is collected using a Cambridge filter pad and determined gravimetrically. The pad is combined with the impinger solution and fortified with internal standard. Analysis is based on CORESTA Recommended Method CRM No. 84 (ISO 24199) using internal standard quantitation. Propylene Glycol, Glycerin, Nicotine, Menthol and Water are reported as mg of analyte per unit of consumable.(i.e., one stick or one capsule)

Aerosol Collection

Figure 1: Modified Sample Trap (CFP + Impinger) for Collecting aerosol emissions from HTPs



Sample Preparation



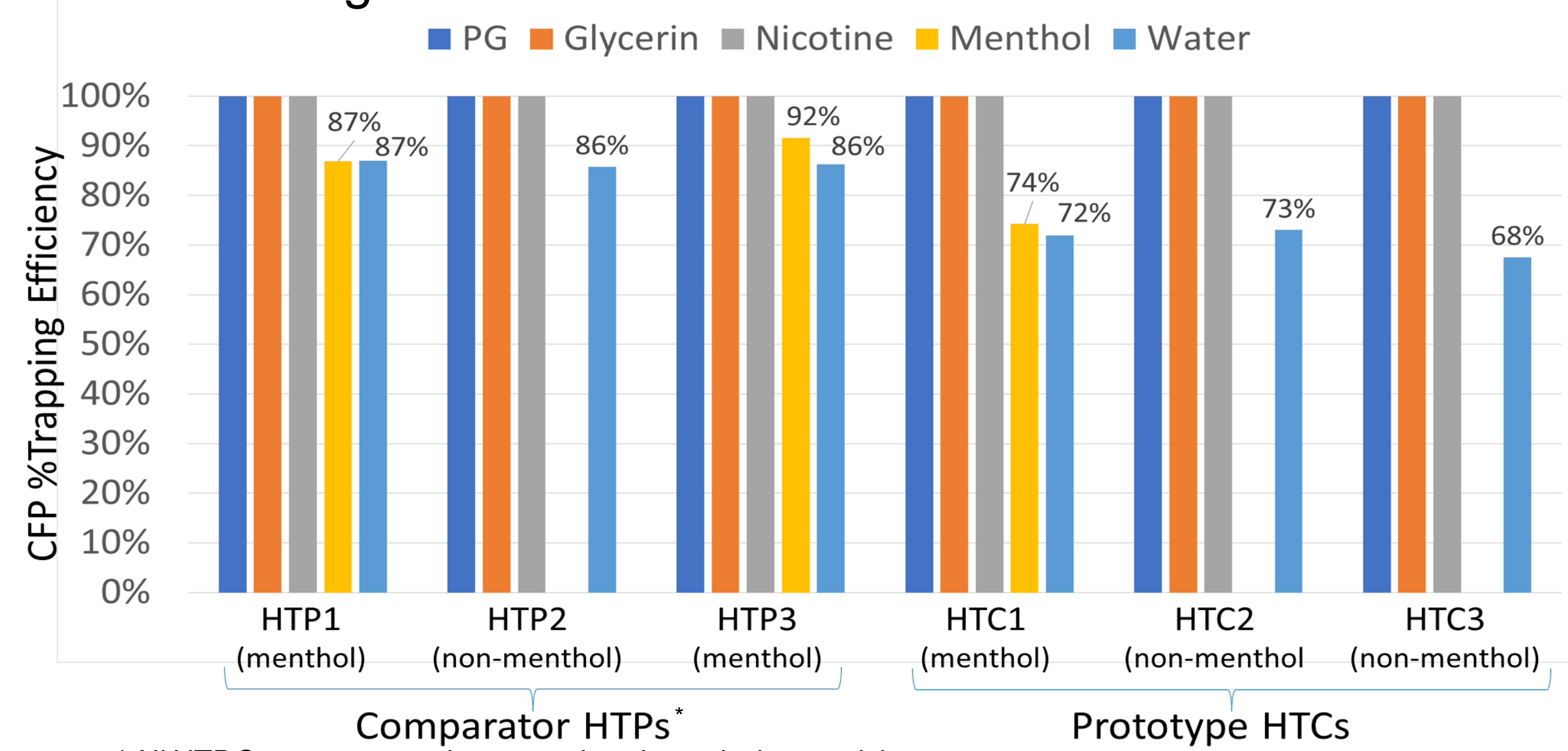
Analytical Method using GC - FID / TCD

Parameters	Description	Parameters	Description
Instrument	Agilent 7890B GC - FID	Instrument	Agilent 7890B GC - TCD
Injection Volume	1.0 µL	Injection Volume	2.0 µL
Injection Mode	Split (100:1)	Injection Mode	Split (20:1)
Injection Port Temperature	250 °C	Injection Port Temperature	225 °C
GC Columns	DB-ALC1 15m x 0.32mm ID x 1.8µm df	GC Columns	DB-ALC1 30m x 0.32mm ID x 1.8µm df
Flow Rate (Constant Flow)	1.5 mL/min	Flow Rate (Constant Flow)	1.5 mL/min
Oven Program	90 °C hold for 1 min, 15 °C /min to 120 °C hold for 1 min, 30 °C /min to 280 °C, hold for 4 min	Oven Program	90 °C hold for 1 min, 15 °C /min to 120 °C hold for 1 min, 30 °C /min to 280 °C, hold for 4 min
FID temperature	275 °C	TCD temperature	250 °C
Run time	13.3 min	Run time	13.3 min

Modified CRM No.84 (ISO 24199) has been validated to quantitate Propylene Glycol, Glycerin, Nicotine, Menthol and Water in aerosol emissions from HTPs

Results–Trapping Efficiency with CFP Only

Breakthrough of water and menthol from CFP was observed [†]



* All HTP Comparator products tested use heated tobacco sticks
[†] Trapping Efficiency determined by separately measuring the CFP followed by two impingers in series

Validation Summary

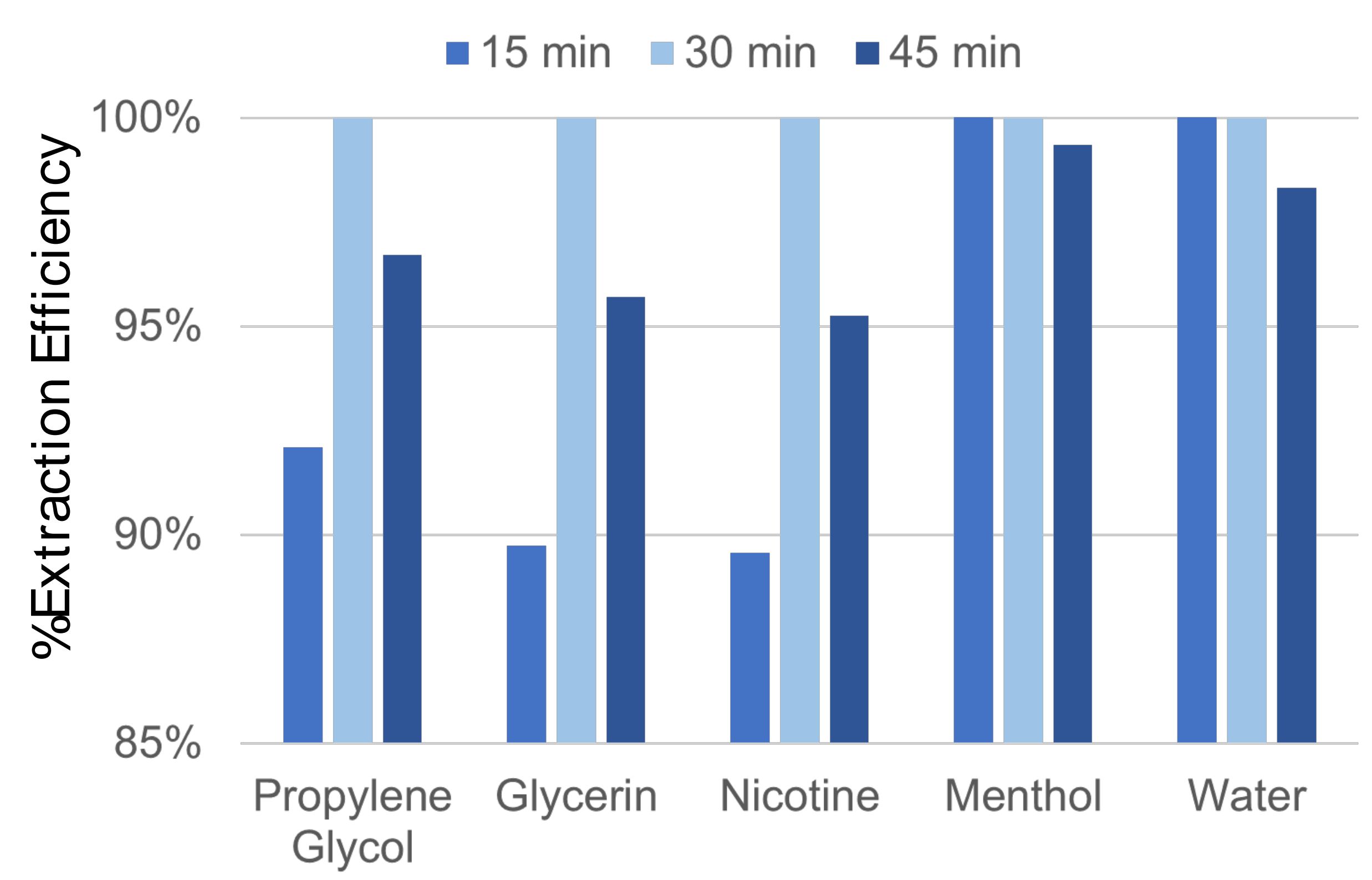
Parameter	Propylene Glycol	Glycerin	Nicotine	Menthol	Water
Calibration Range (mg/mL)	0.050 – 5.0	0.10 – 5.0	0.025 – 1.0	0.025 – 1.0	0.50 – 10.0
Linearity R ²	≥ 0.999	≥ 0.999	≥ 0.999	≥ 0.999	≥ 0.999
Trapping Efficiency (CFP ¹ + Impinger) ² (n=3)	$\geq 99^1$	$\geq 99^1$	$\geq 99^1$	$\geq 99^2$	$\geq 97.8^2$
Laboratory Fortified Matrix %Recovery (Intense, n=3)	96 – 97	102.4 – 102.8	86 – 96	89.7 – 90.5	94.6 – 98
Laboratory Fortified Blank %Recovery (n=3)	100.3 – 100.6	96.4 – 97.9	99.9 – 100.7	96.5 – 96.9	103.1 – 105.9
Repeatability %RSD (Intense, n=6)	≤ 12.9	≤ 14.3	≤ 14.6	≤ 8.5	≤ 10.5
3-day Intermediate Precision %RSD (Intense, n=18)	≤ 7.6	≤ 9.0	≤ 8.8	≤ 5.2	≤ 8.2
Repeatability %RSD (Non-Intense, n=6)	≤ 9.2	≤ 12.4	≤ 10.0	≤ 3.8	≤ 7.1
3-day Intermediate Precision %RSD (Non-Intense, n=18)	≤ 7.1	≤ 8.1	≤ 6.8	≤ 4.2	≤ 6.8
Limit of Quantitation (LOQ) (mg/unit)	0.33	0.67	0.17	0.17	3.33

References

- CORESTA Recommended Method No. 84, *Determination of Glycerin, Propylene Glycol, Water, and Nicotine in the Aerosol of E-Cigarettes by Gas Chromatographic Analysis* (June 2017)).
- CORESTA Recommended Method No. 101, *Definitions And Standard Conditions: Aerosol Generation And Collection For Electrically Heated Tobacco Products* (February 2023)
- ISO 20778:2018, *Cigarettes — Routine analytical cigarette smoking machine — Definitions and standard conditions with an intense smoking regime*
- ISO 3308:2012, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

Results–Extraction Time Study

30 min extraction time and no more than 45 min



Results (In Aerosol Emissions)

Sample	Aerosol Mass (mg/unit)	PG (mg/unit)	GLY (mg/unit)	Menthol (mg/unit)	Nicotine (mg/unit)	Water (mg/unit)
Comparator HTP (non-menthol)*	51.8	0.433	4.81	BLOQ	1.32	43.9
Comparator HTP (menthol)*	43.7	0.25	4.02	2.01	1.22	46.4
Prototype HTC (non-menthol)	42.0	6.11	10.2	BLOQ	1.16	24.8
Prototype HTC (menthol)	39.6	1.83	11.4	1.72	1.71	23.5

* All HTP Comparator products tested use heated tobacco sticks

Conclusions

- All pre-determined acceptance criteria were met during the method validation
- This method was validated and determined to be fit for purpose to quantitatively determine five principal components in aerosol emissions from HTPs.
- Modifications to the sample trap to include a pad followed by an impinger with a fritted tip containing 20 mL Isopropanol (low water, < 0.015%); improved trapping efficiency beyond a pad only collection, to allow for the accurate reporting of menthol and water from HTP emissions.

