Real-Time Puff-by-Puff Analysis of E-Cigarette Aerosol using GC-MS

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Background

- E-cigarette formulations and aerosols typically contain propylene glycol (PG), glycerin, water, nicotine and flavors

- There is a need for rapid prototype evaluation of e-cigarette aerosol composition to:
  - Measure formulation components on a puff-by-puff basis
  - Evaluate formulation changes
  - Explore device configuration changes

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Objective

Develop an automated puff-by-puff detection system for real-time analysis of e-cigarette aerosol to better understand how these devices perform during use.

Requirements:

- Real-time whole aerosol analysis
- Require no sample preparation
- Use one e-cigarette device per analysis
- Monitor analytes on a puff-by-puff basis
- Report data to compare prototype aerosols to a control product

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Overview

- System Configuration
  - Whole aerosol collection
- Methodology
  - Puffing/Gas Chromatography Mass Spectrometry (GC-MS) parameters
  - Carry-over between puffs
  - Instrument precision
- Applications:
  - Formulation and device changes
  - Prototype performance during battery life
  - Semi-quantitative analysis of single puff yield

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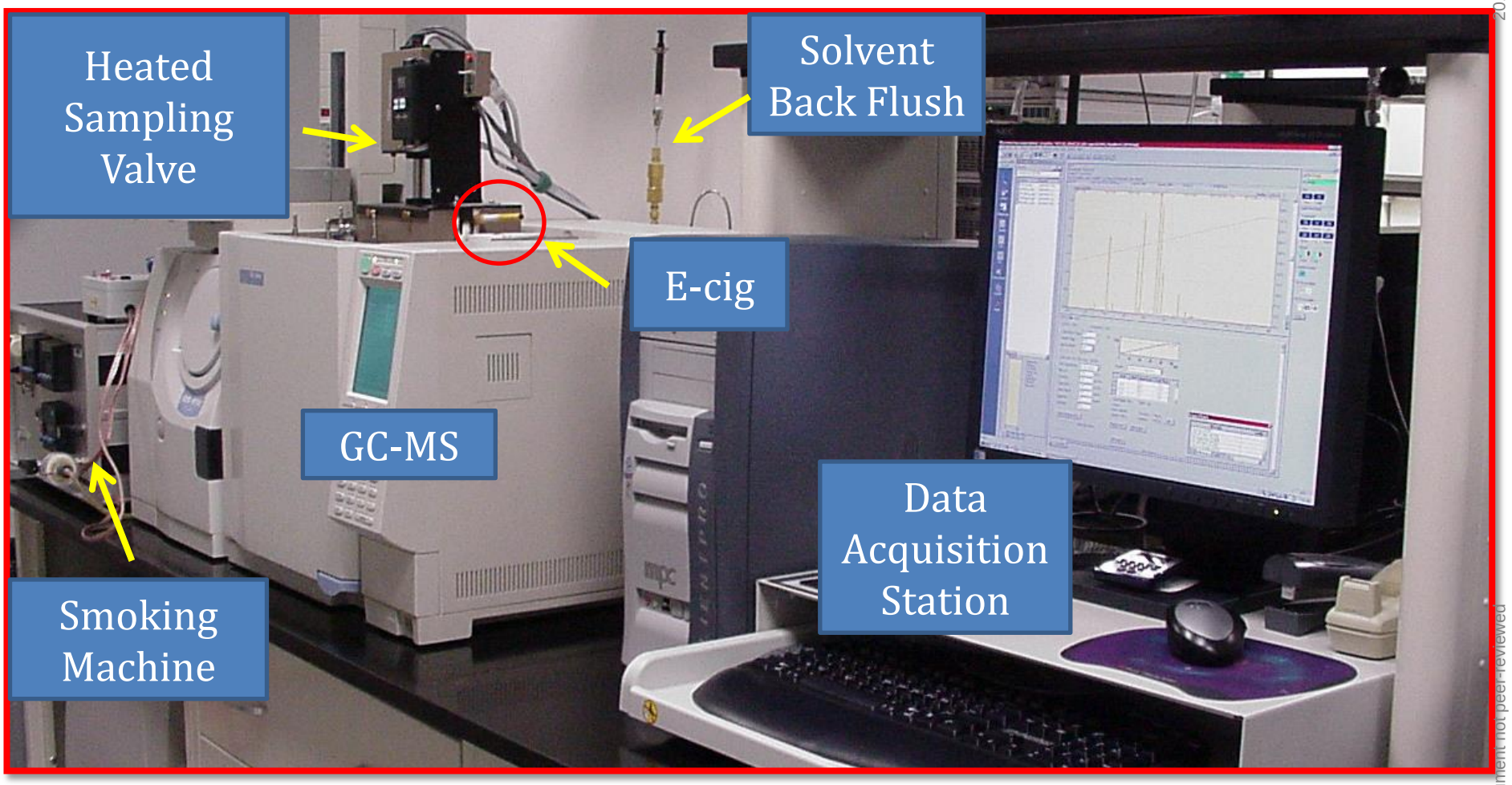
Real-Time Puff-by-Puff GC-MS System



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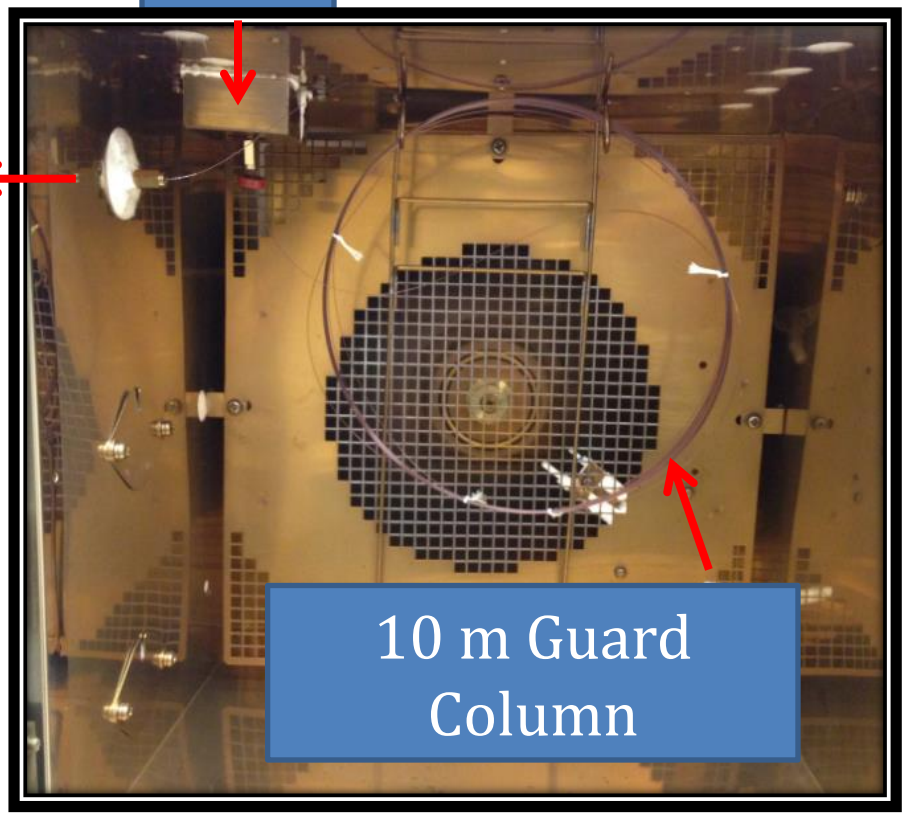

No chromatographic separation and retention of compounds

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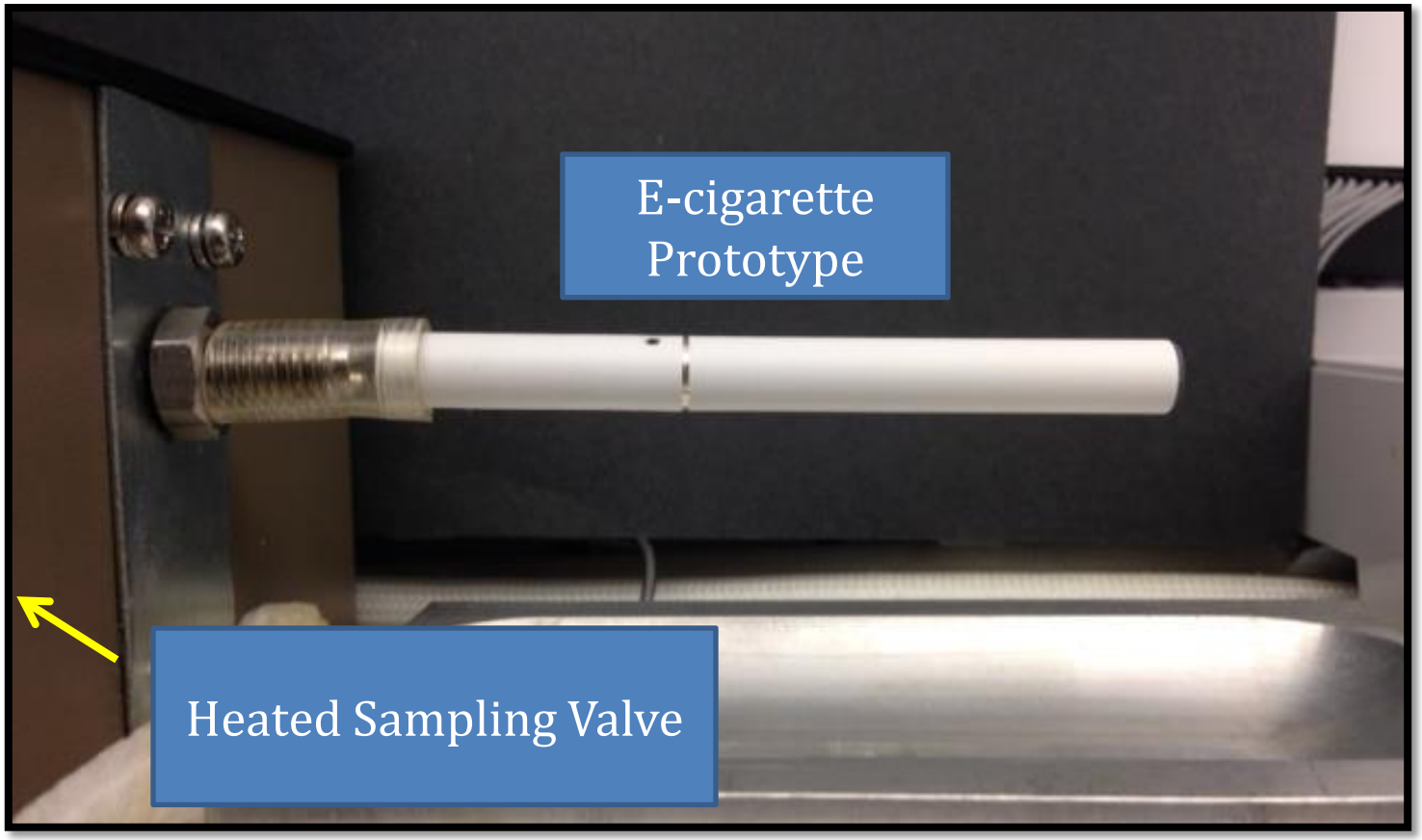
Real-Time Puff-by-Puff GC-MS System



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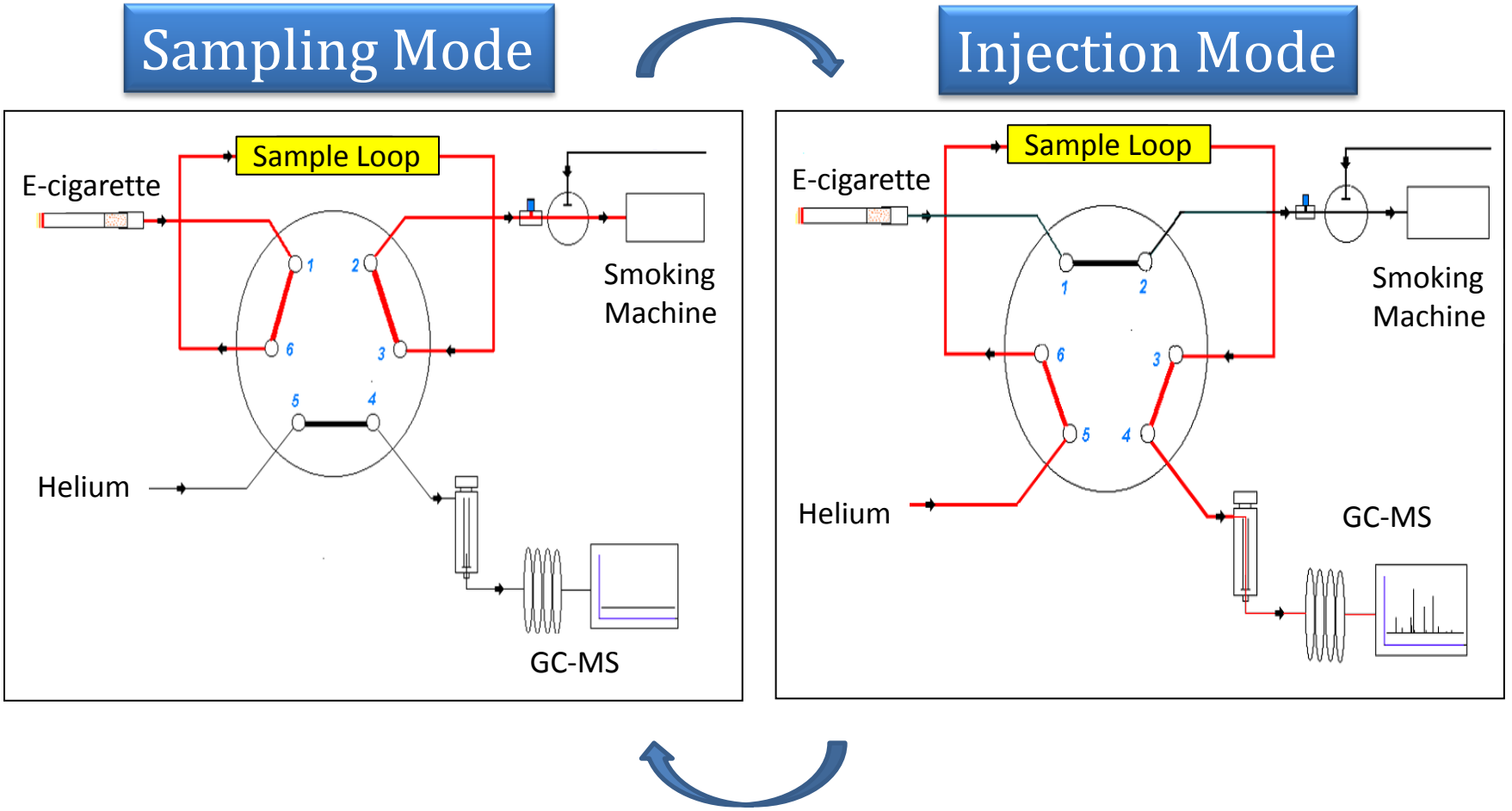
Puff-by-Puff Aerosol Sampling



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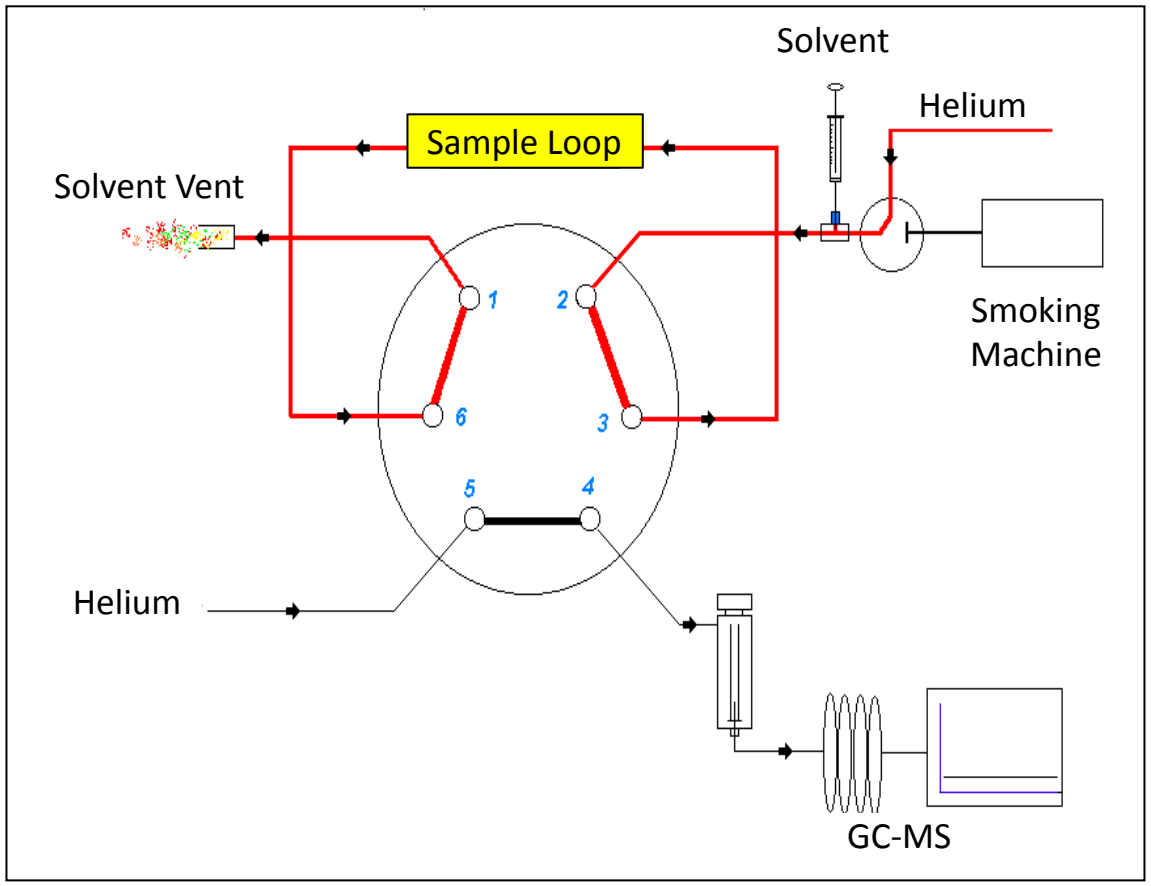
Puff-by-Puff Aerosol Sampling

Back Flush Mode



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Analytical Method

Puffing Parameters:

- Puff volume - 55 mL
- Puff duration - 4 seconds
- Puff frequency - 30 seconds
- Puff profile - Square wave

GC-MS Parameters:

- 2 mL aerosol sample injection
- 450:1 split ratio
- Inlet temperature 220 °C
- Sampling valve at 200 °C
- 10 m x 0.25 mm ID guard column at 250 °C
- Ion source at 240 °C, transfer line at 250 °C

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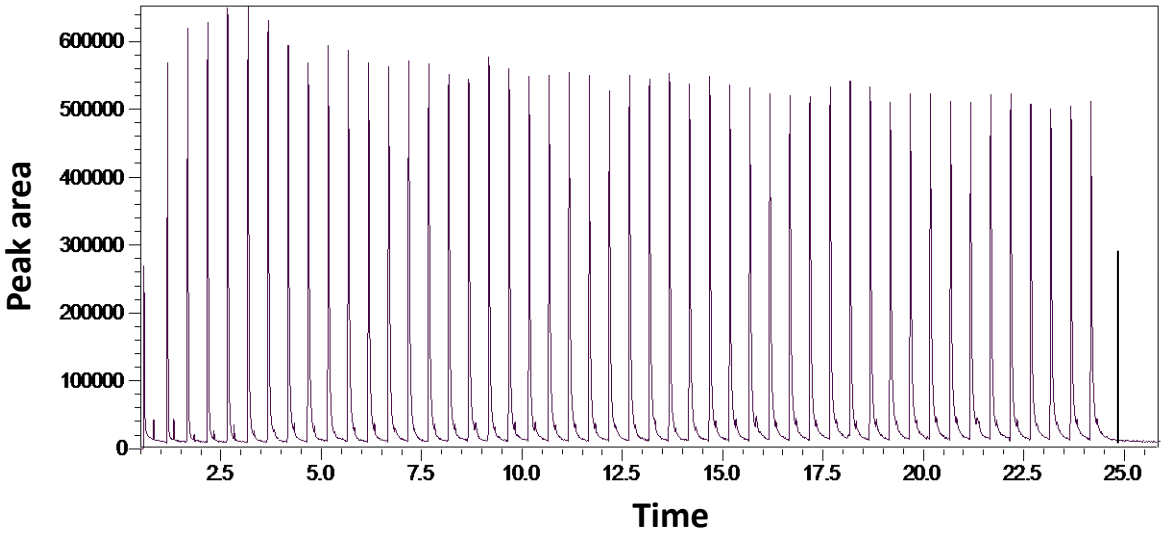
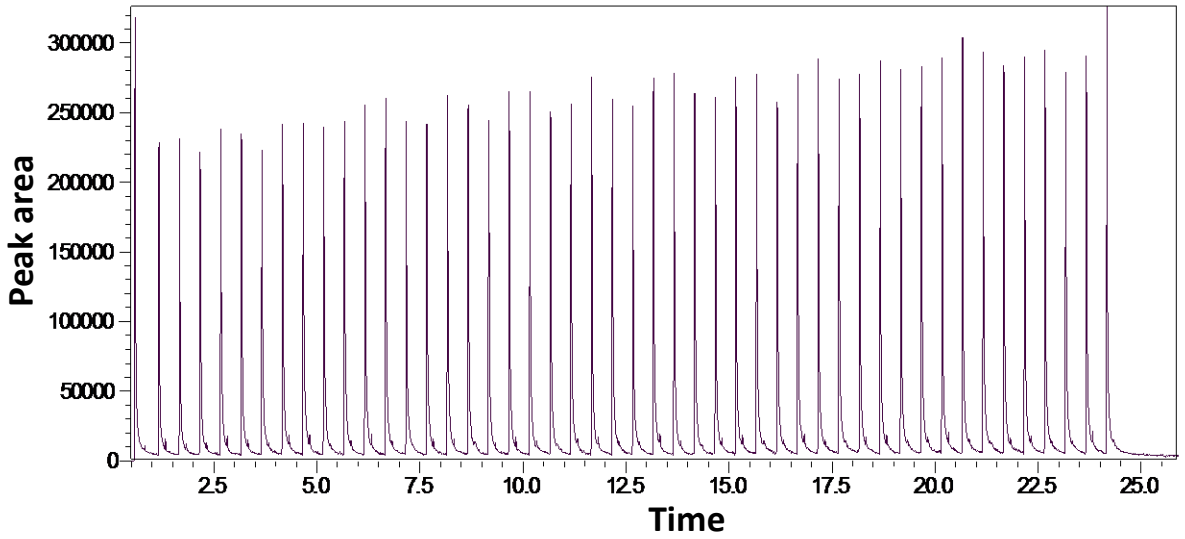
Chromatograms – PG and Glycerin

Puffs 1-50
Propylene Glycol,
m/z=76



Puffs 1-50
Glycerin,
m/z=92



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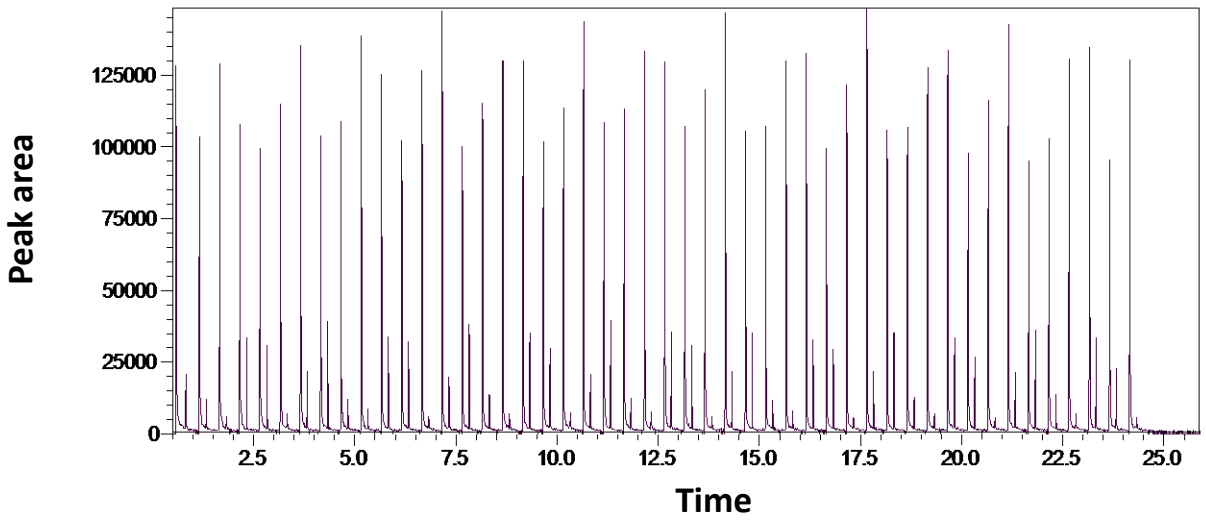
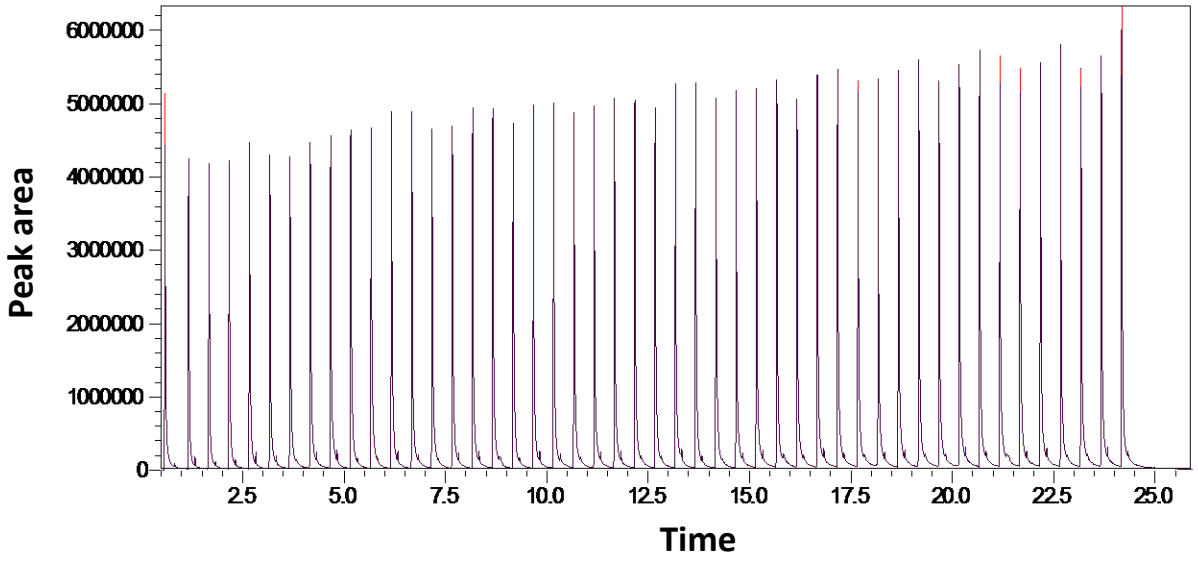
Chromatograms - Nicotine and Menthol

Puffs 1-50 Menthol, m/z=138



Puffs 1-50 Nicotine, m/z=84



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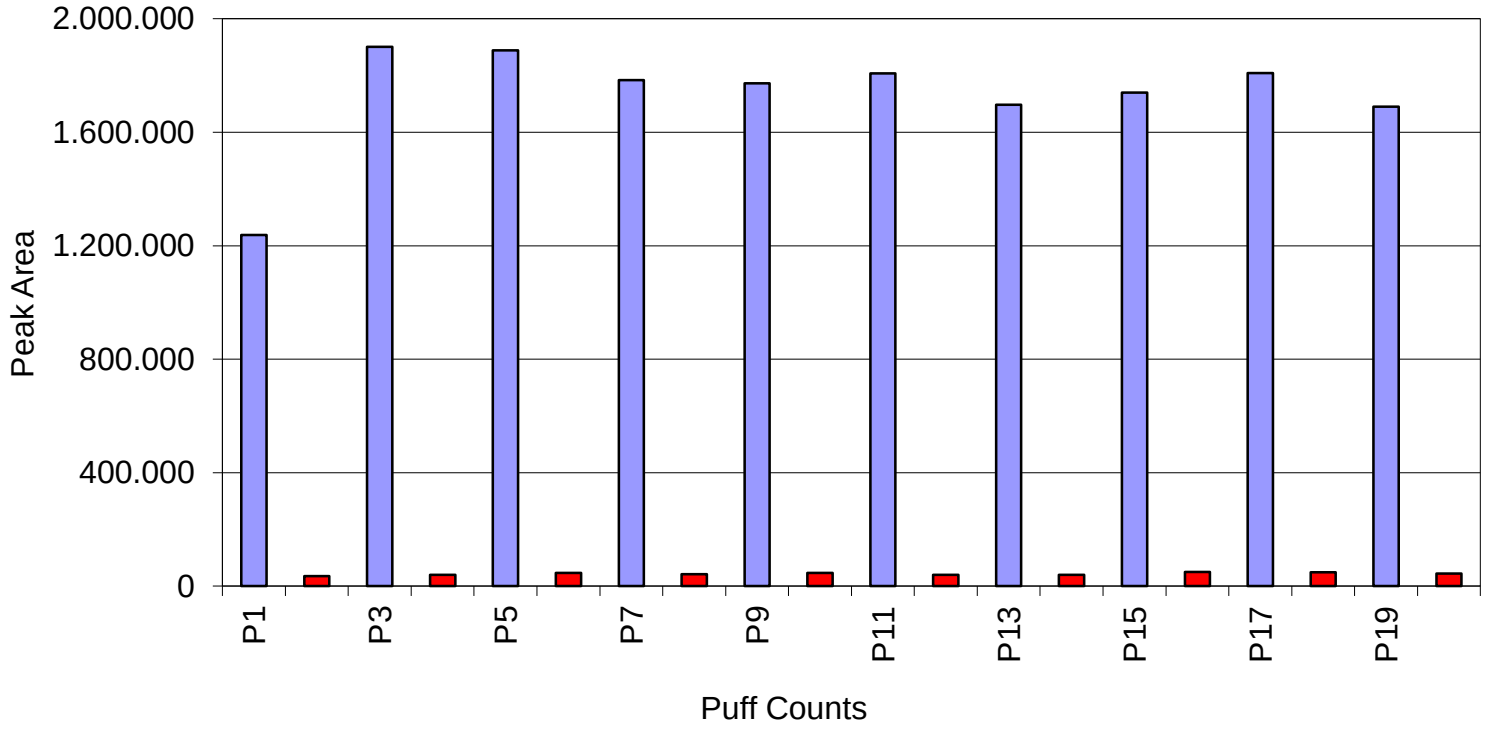
Nicotine Carry-Over Between Puffs



Note: The red bars are the puffs without e-cigarette

Low nicotine carry over (<3%) observed between puffs

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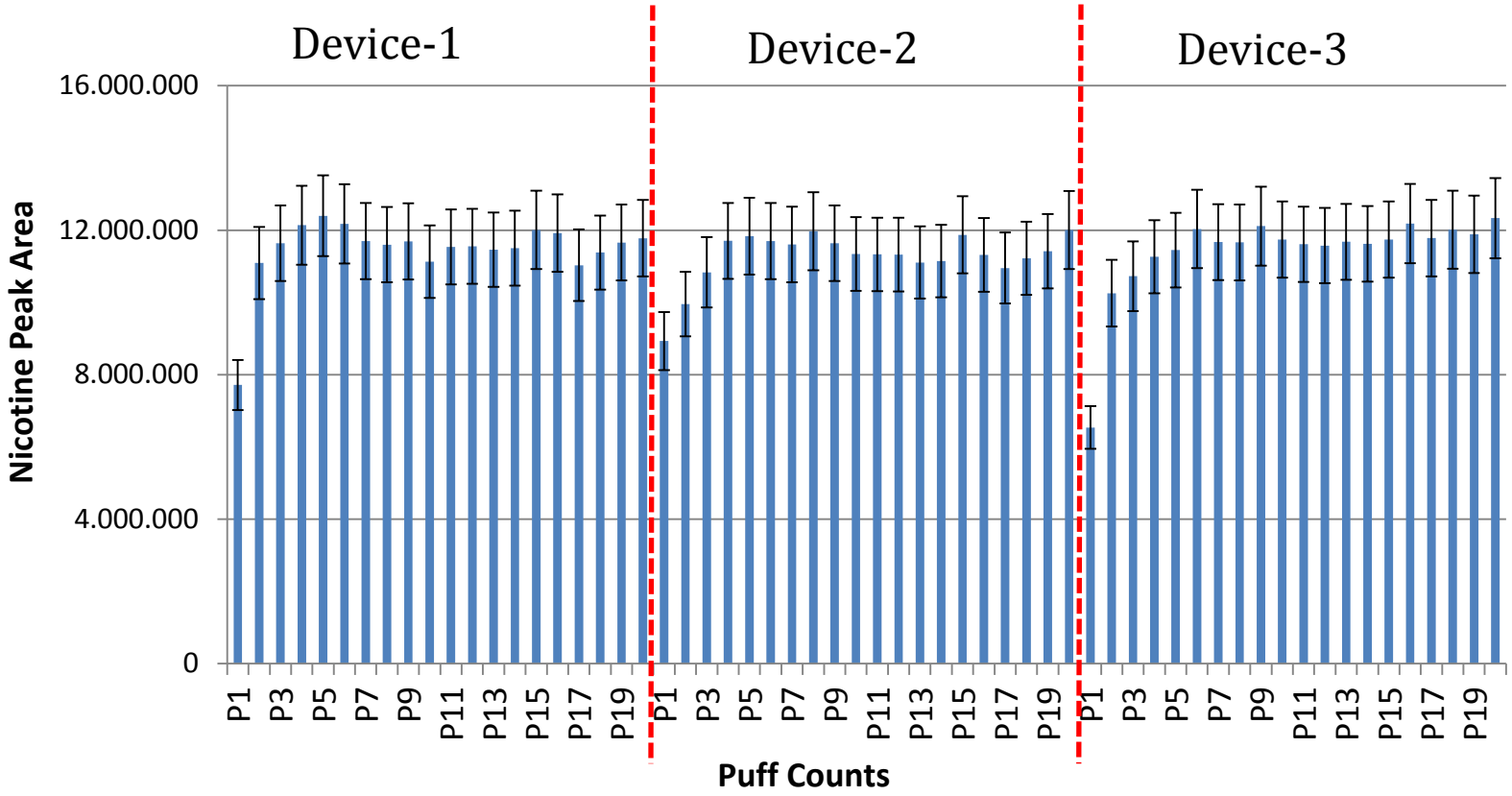
Puff-by-Puff Nicotine – Method Precision



The puff-by-puff method precision (%RSD) is < 10%

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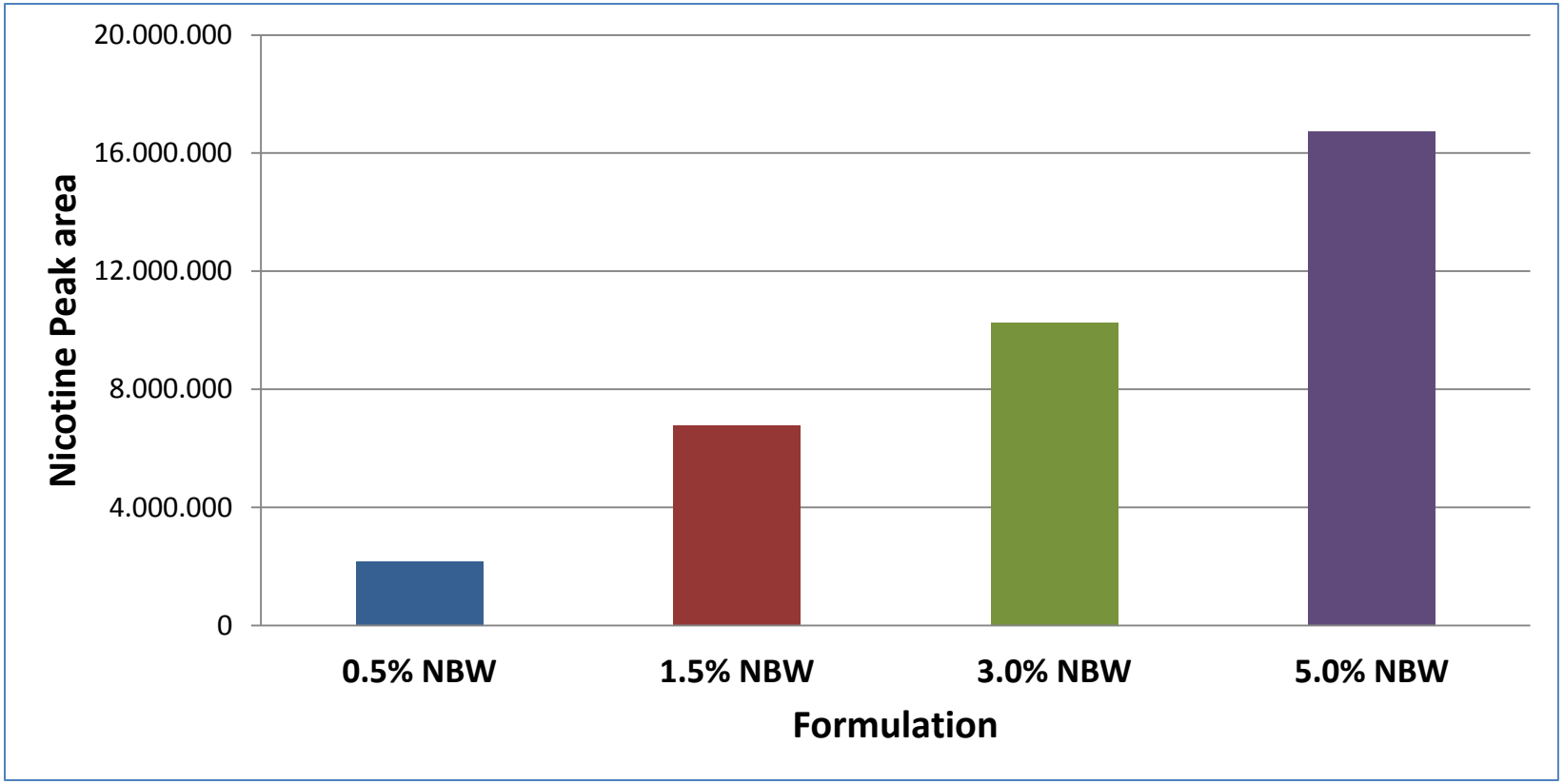
Formulation Change: Cumulative Nicotine (1-20 puffs, N=2)



Note: A single device was used with different nicotine loading

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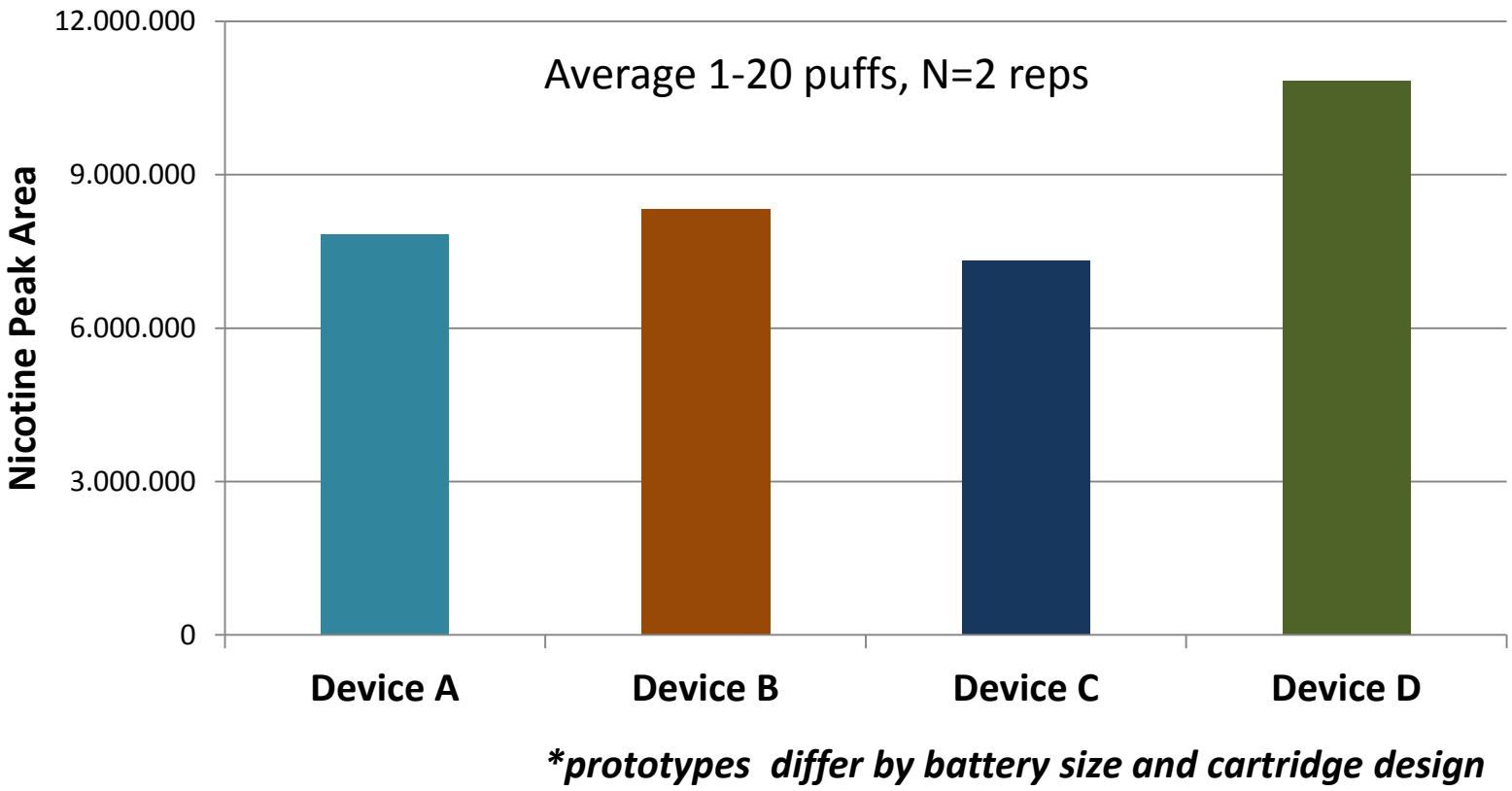
Device Change: Puff-by-Puff Nicotine Profile
Different Prototypes/Same Formulation



prototypes differ by battery size and cartridge design

Same formulation in different devices results in varying yields

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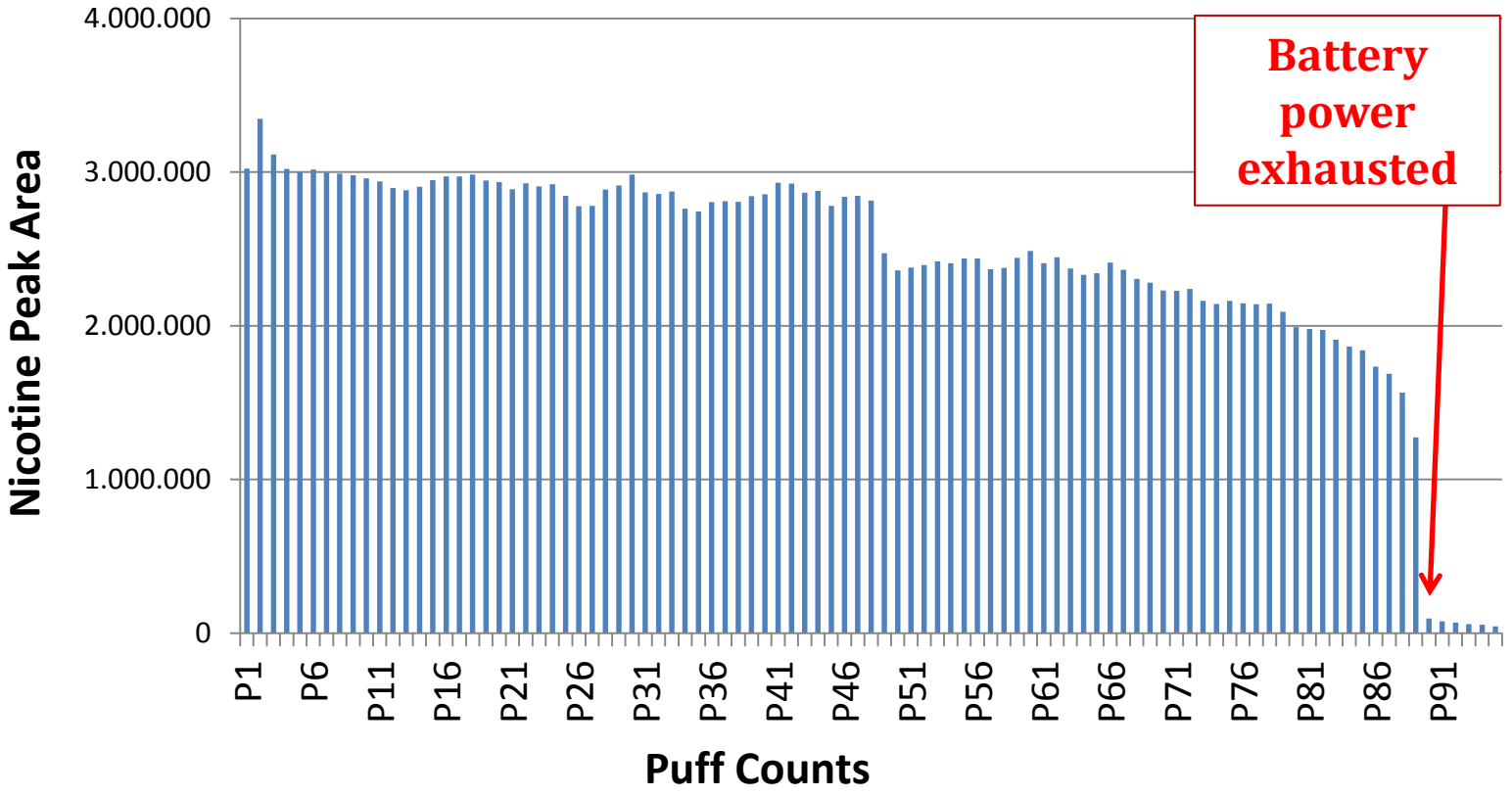
Prototype Analysis -Puff-by-Puff Nicotine
Charged Battery and New Cartridge



Approximately 90 puffs delivered using this prototype device

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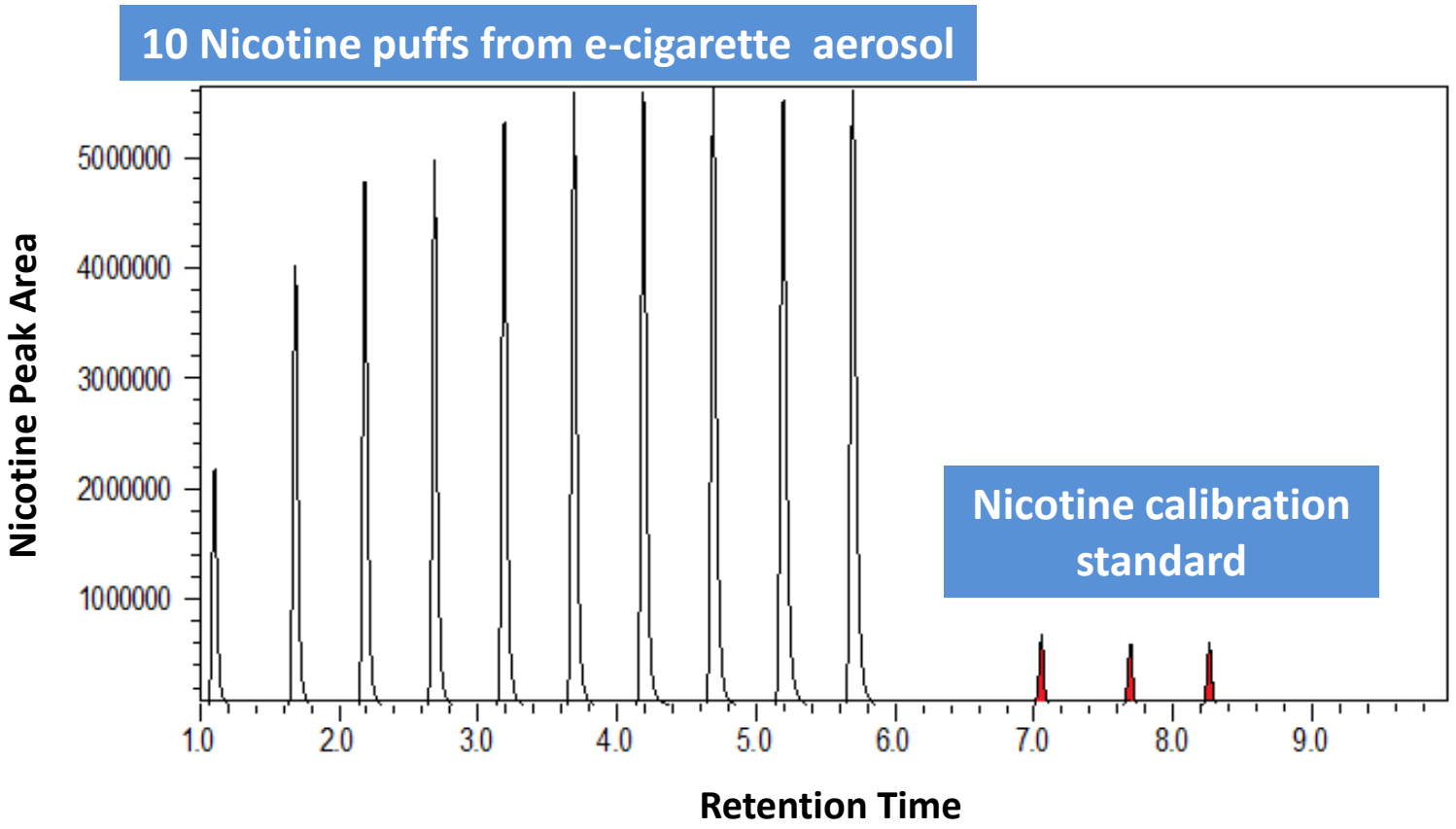
Single Puff Nicotine Yield



Nicotine yield can be estimated using a single point calibration

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Summary

- Puff-by-puff GCMS is a rapid screening method for e-cigarette prototype characterization
- The automated real-time method offers high selectivity, sensitivity and precision for measurement of e-cigarette aerosol composition using a limited number of prototypes
- The method is capable of monitoring formulation components in multiple puffs of e-cigarette whole aerosol

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