Infrared Thermal Imaging Applied to E-Cigarette Heater Core Temperature Measurement

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Introduction

- FDA PMTA Draft Guidance for ENDS* recommends providing information on “design parameters that would be affected by and that would affect aerosolizing apparatus performance”
- Characterization of device temperatures is important, as changes in temperature can result in changes in aerosol ingredients
- It is difficult to accurately measure temperature in ENDS devices using standard methodologies (e.g. thermocouples)

*FDA, May 2016. Premarket Tobacco Product Applications for Electronic Nicotine Delivery Systems, Guidance for Industry, Draft Guidance. Page 39, line 1569-1571, Food and Drug Administration, Rockville, MD.

Limitations of Thermocouple (TC) Heater Core Measurement

- Difficult to position TC in heater core (heater assembly)
  - TC's are Fragile
  - Time consuming to insert TC and prevent movement
  - Location reproducibility of the TC is a problem

- TC is single point measurement

- TC mounted in heater assembly acts as a heat sink to surrounding areas

- Presence of TC in heater assembly may disturb airflow and interfere with wicking

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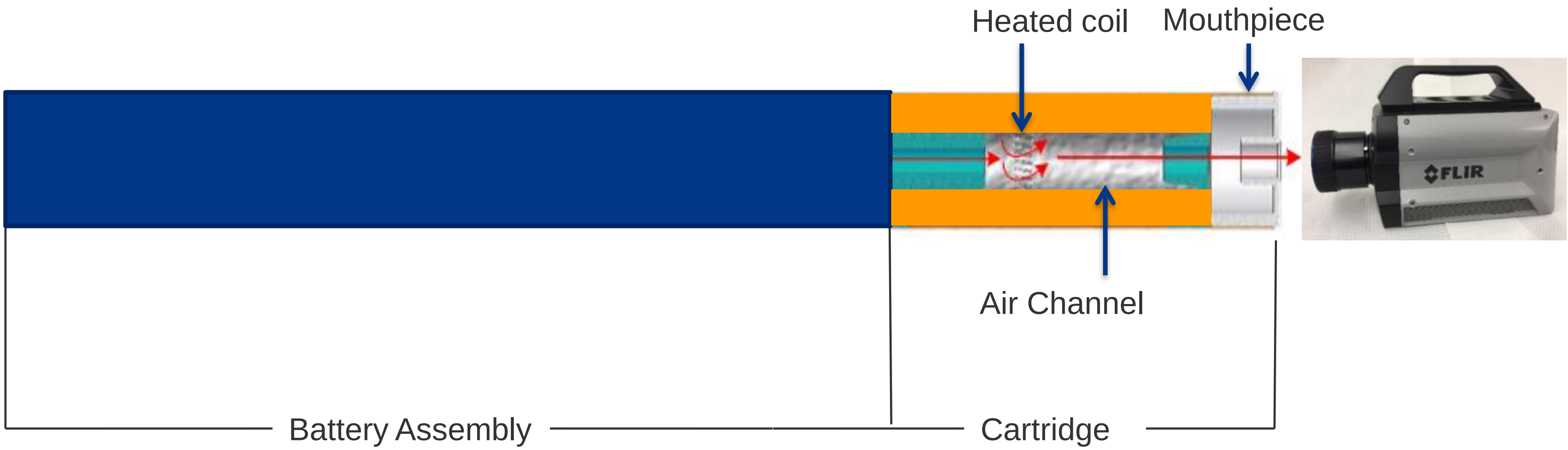
FLIR® Imaging

- Profiles entire heater core
- Realtime measurement
  - Fast response time (30 frames per second)
  - Captures the hottest point of a puff (i.e. hottest frame)
- Capable of auto triggering
- Labview automation for pump puff activation and FLIR® activation
  - Any sequence of puffs can be recorded
  - Puff duration and volume can be changed

ENDS Schematic

Infrared thermal imaging through the front of the cartridge



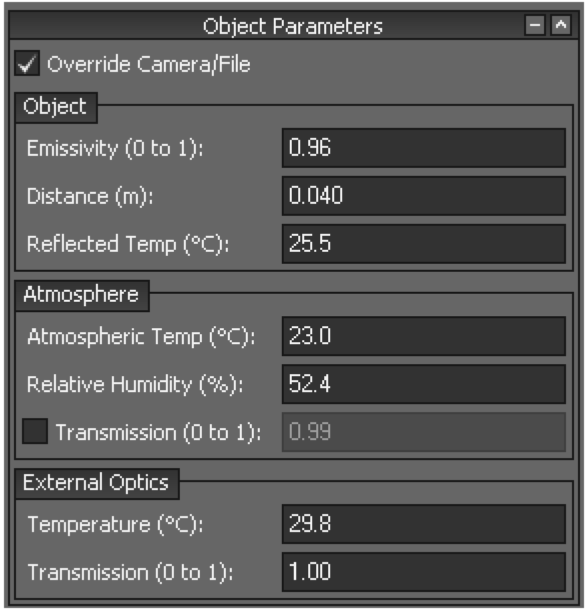
Optical Resolution – FLIR® E-cigarette Setup

- Optimized optical resolution allows for high spatial resolution of temperature measurements
  - 45µm/pixel

- Working distance = 0.04m
  - Using 0.5in extension ring
  - 25mm Lens
  - Focal plane array detector pixels (frame size) = 640x512

Emissivity Determination

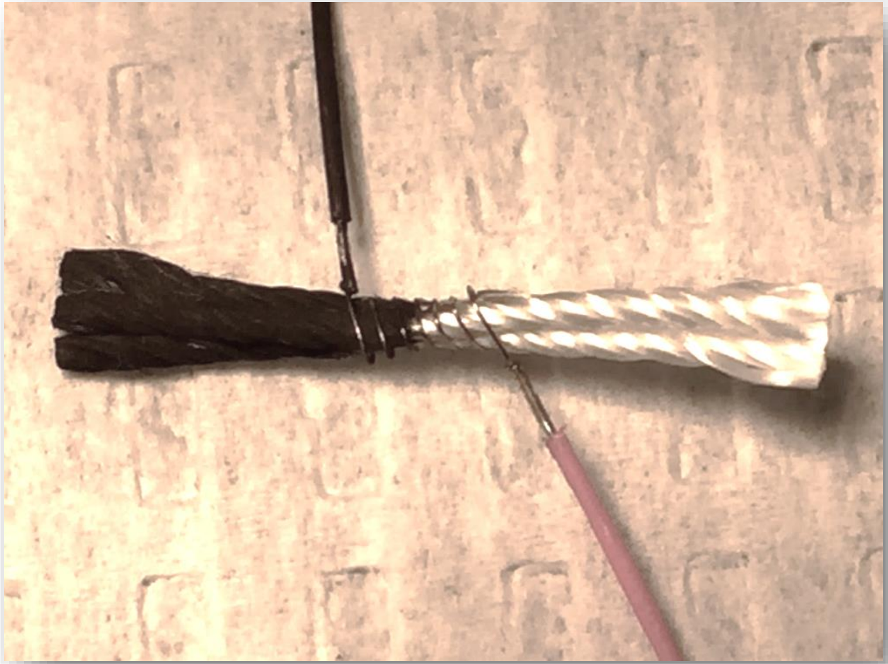
- IR cameras (FLIR®) measure radiance
  - FLIR® calibrations convert radiance to temperature
  - Uses known constants and input parameters
    - Emissivity
    - Reflected temperature (environment)
    - Working distance
    - Ambient air temperature
    - Relative Humidity
    - External optics temperature



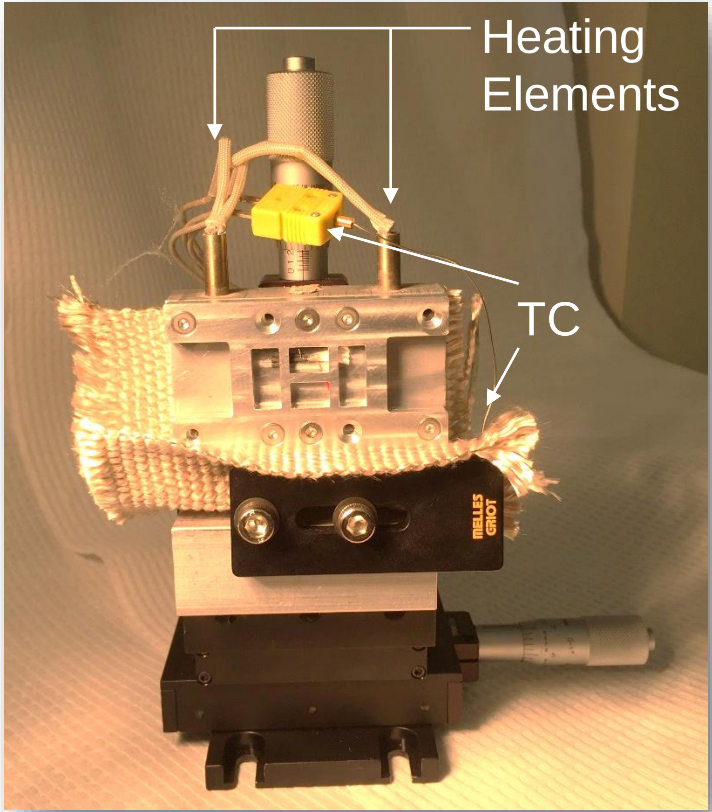
- Emissivity is the most critical parameter
  - Opaque target objects
    - Radiance = Emissivity + Reflectivity

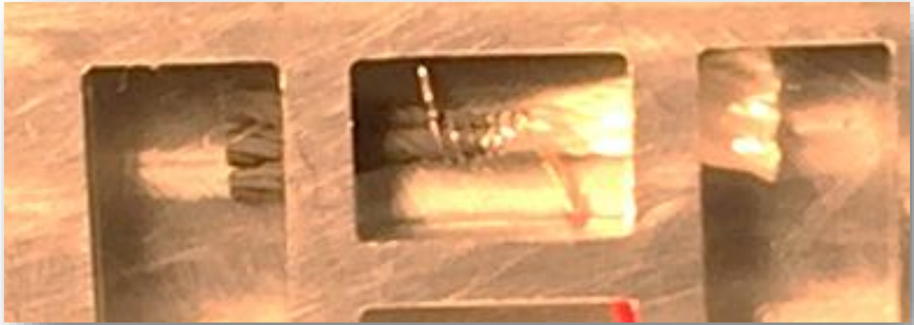
Emissivity Determination

- ASTM E1933-14, "Standard Practice for Measuring and Compensating for Emissivity Using Infrared Imaging Radiometers," 2014.
- Surface modification
  - Heater core is half painted with a high emissivity paint
  - Black Header Paint (704-1093°C, Flame Proof, Silica-Ceramic Coating), recommended by FLIR®
  - Emissivity = 0.99



Emissivity Determination



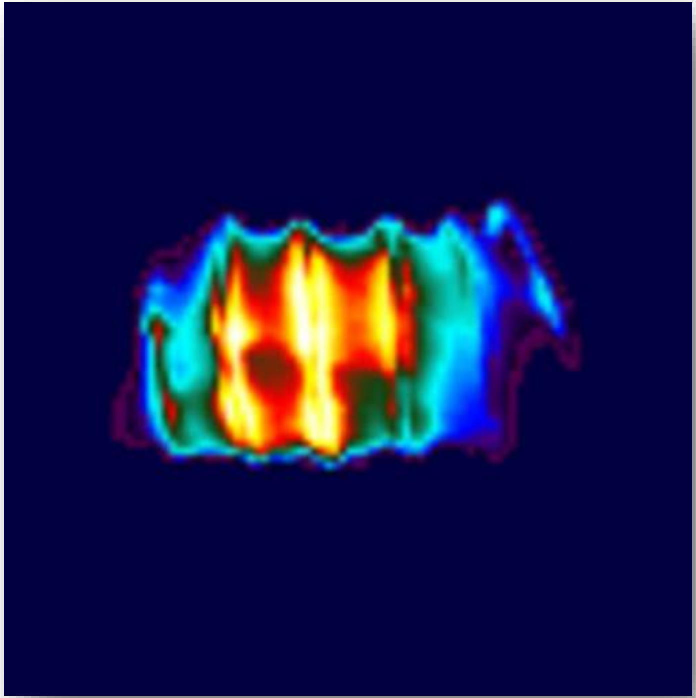


- Custom built emissivity fixture
- Temperature controlled aluminum block
- Allows heater core materials to be mounted securely in direct contact with block
- TC and heating elements are connected to a temperature controller

Heater Core Temp Measurement

Atomizer Cross-sectioned to expose heater core

FLIR® Image of a heater core



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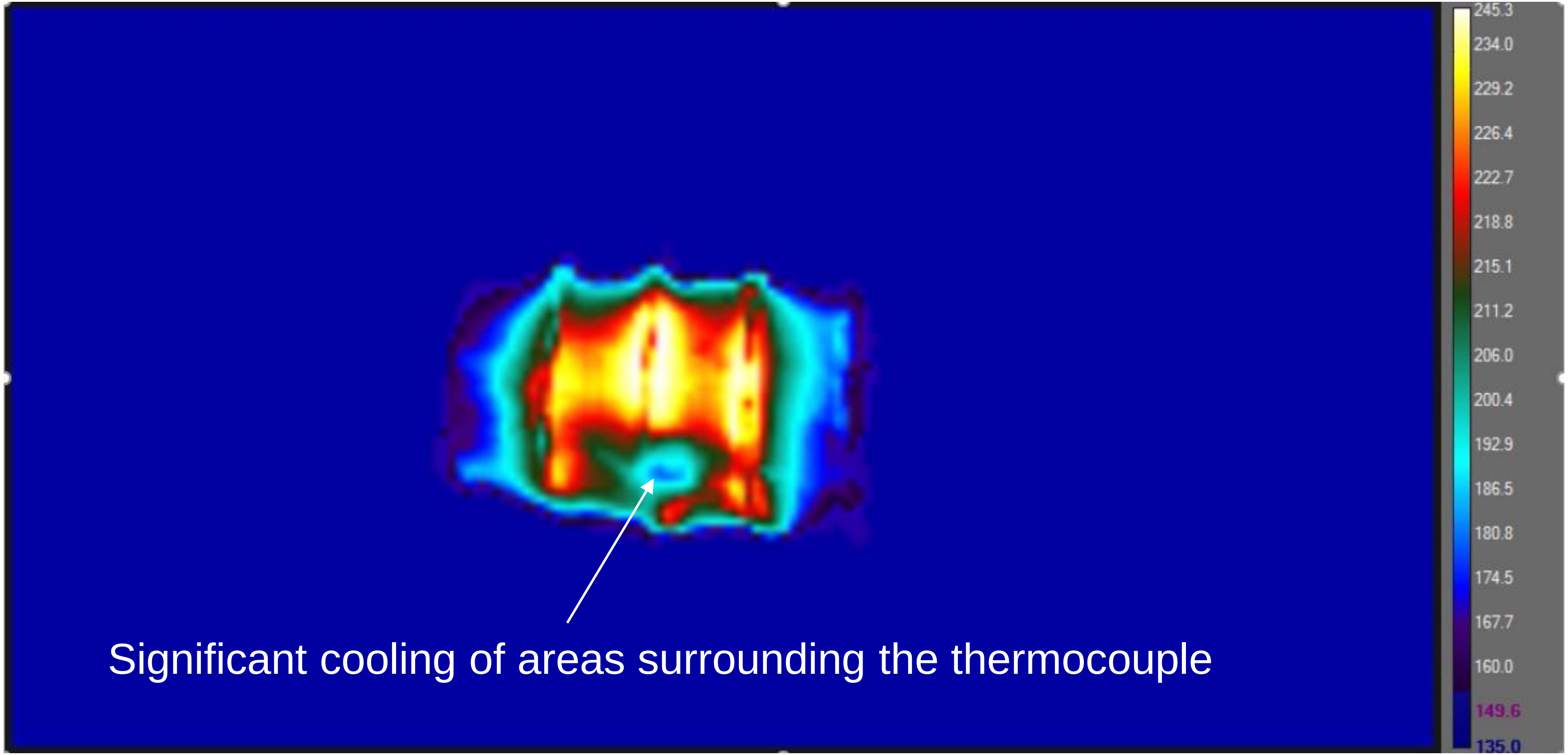
FLIR® Movie – Puff Duration (5s) & Volume (55cc), w/ TC Mounted



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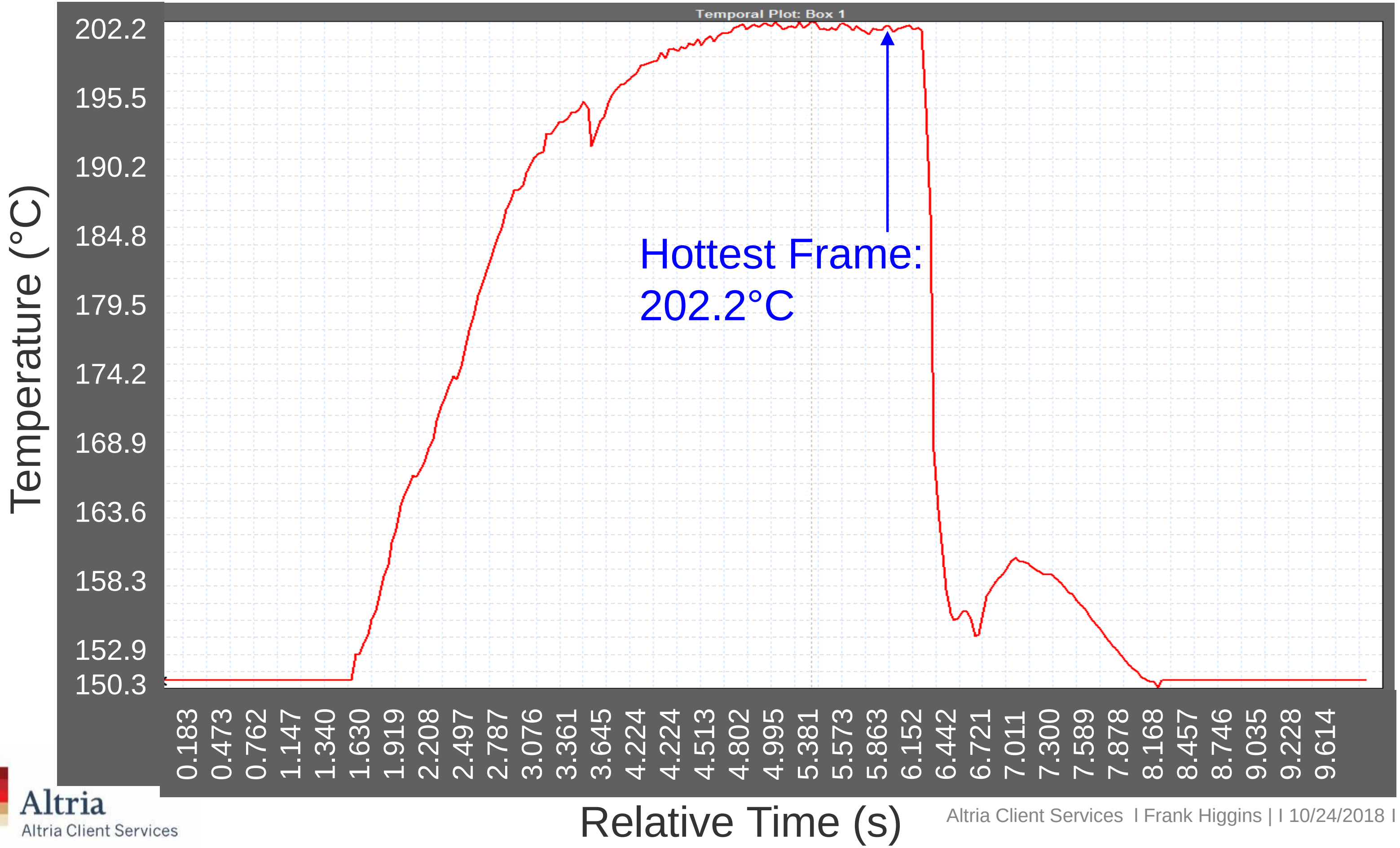
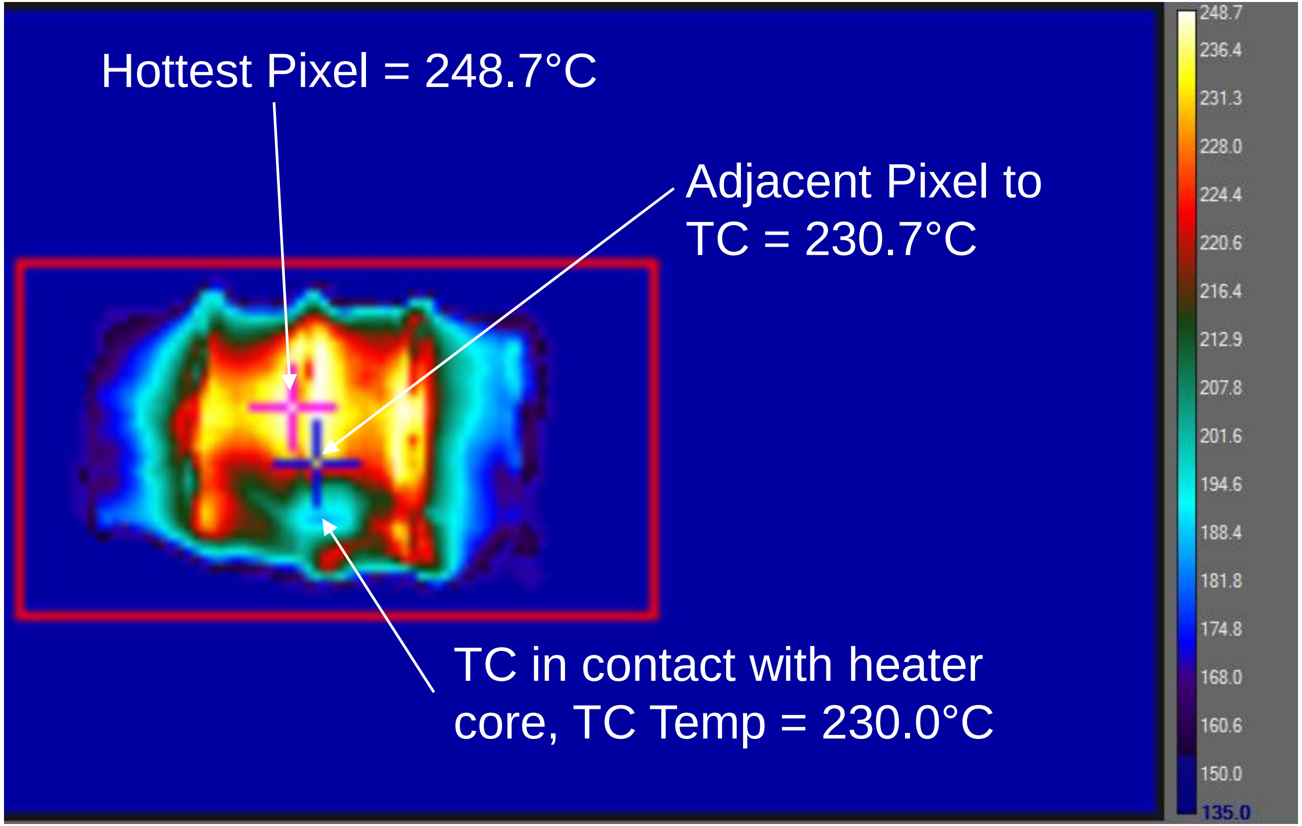
Temporal Plot – Puff Duration (5s) & Volume (55cc), w/ TC Mounted



Hottest Frame #161 – TC Mounted

- TC temperature agrees with FLIR® Mean Temperatures
  - Pixels adjacent to the TC
- TC does not measure the hottest temperatures
  - Heat sinking
  - Positioning



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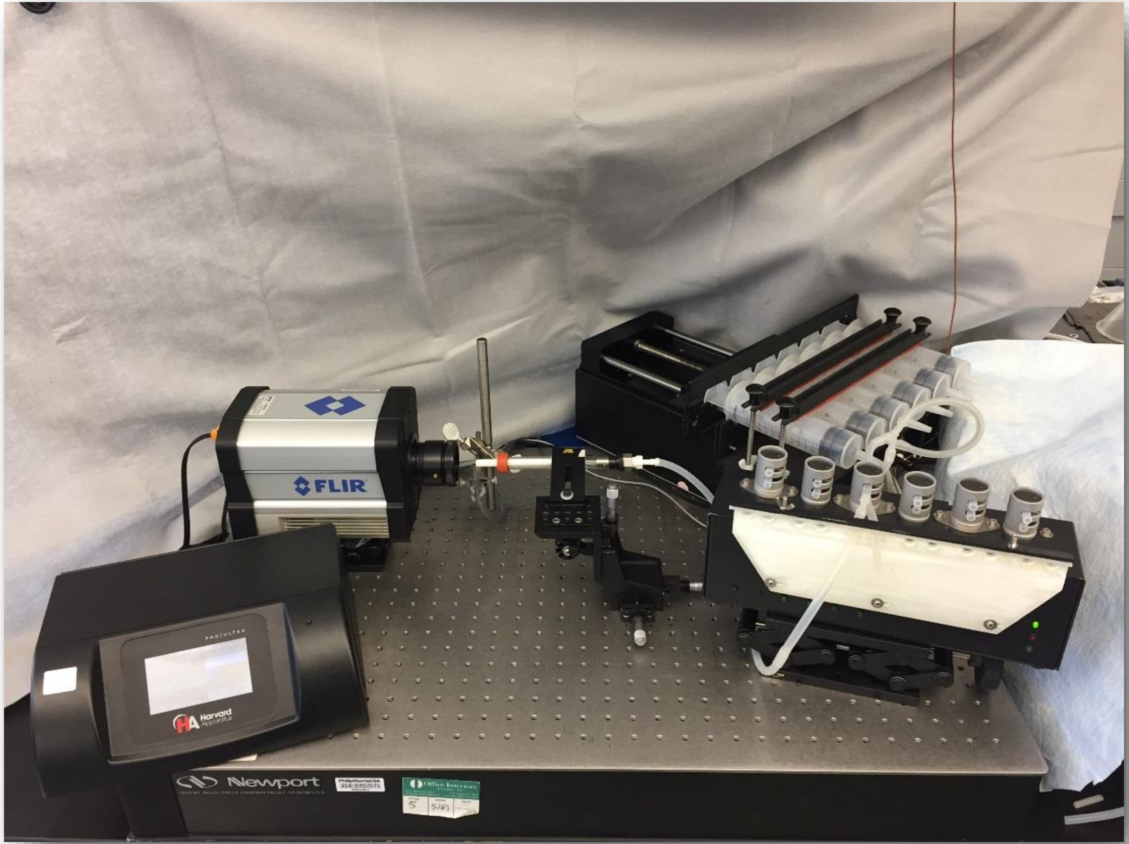
Apparatus Setup – FLIR® w/ Pump



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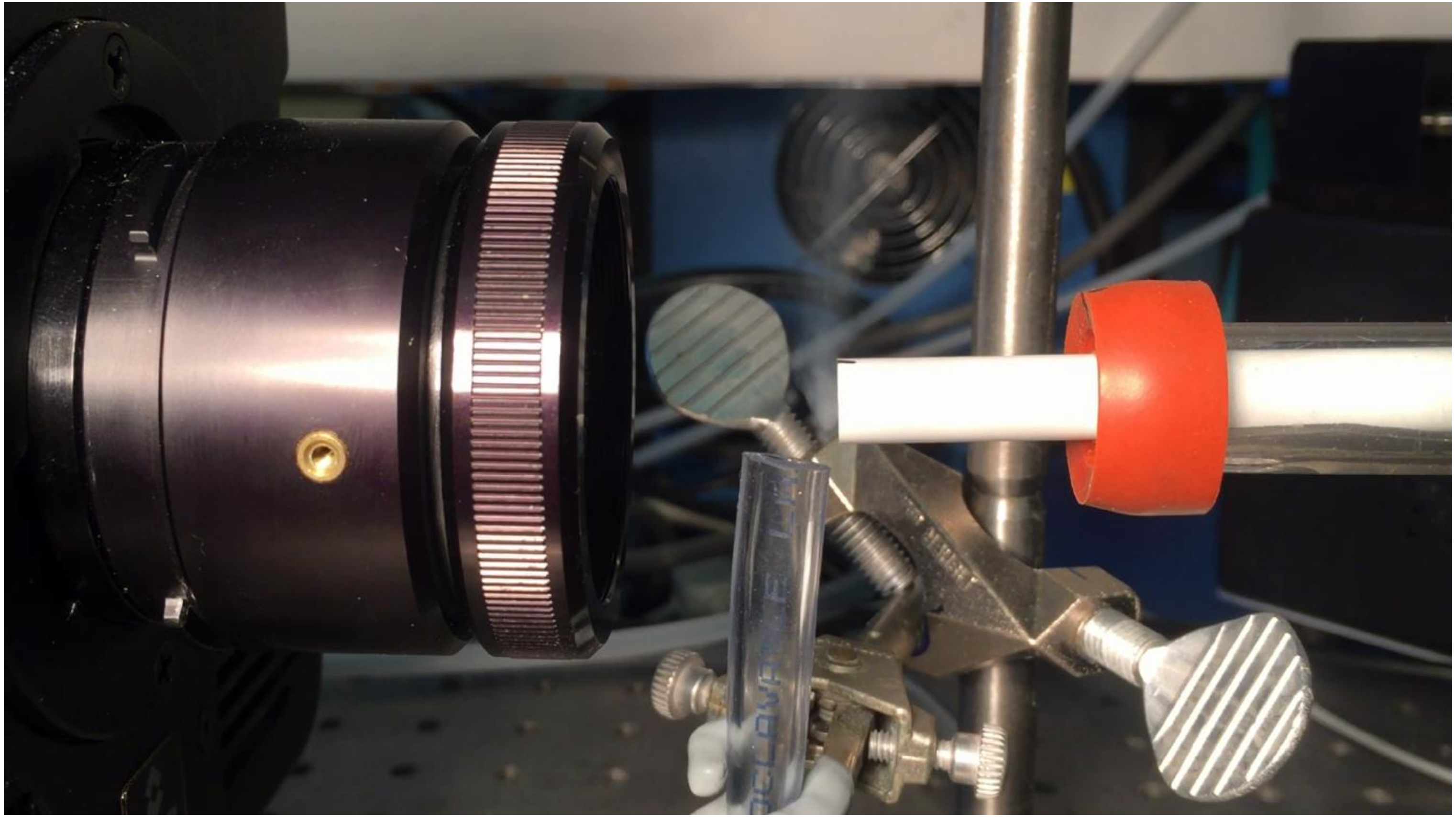
Setup for FLIR® Imaging

Stream of nitrogen gas is used to prevent condensation of vapors onto the FLIR® lens



FLIR® Imaging Parameters

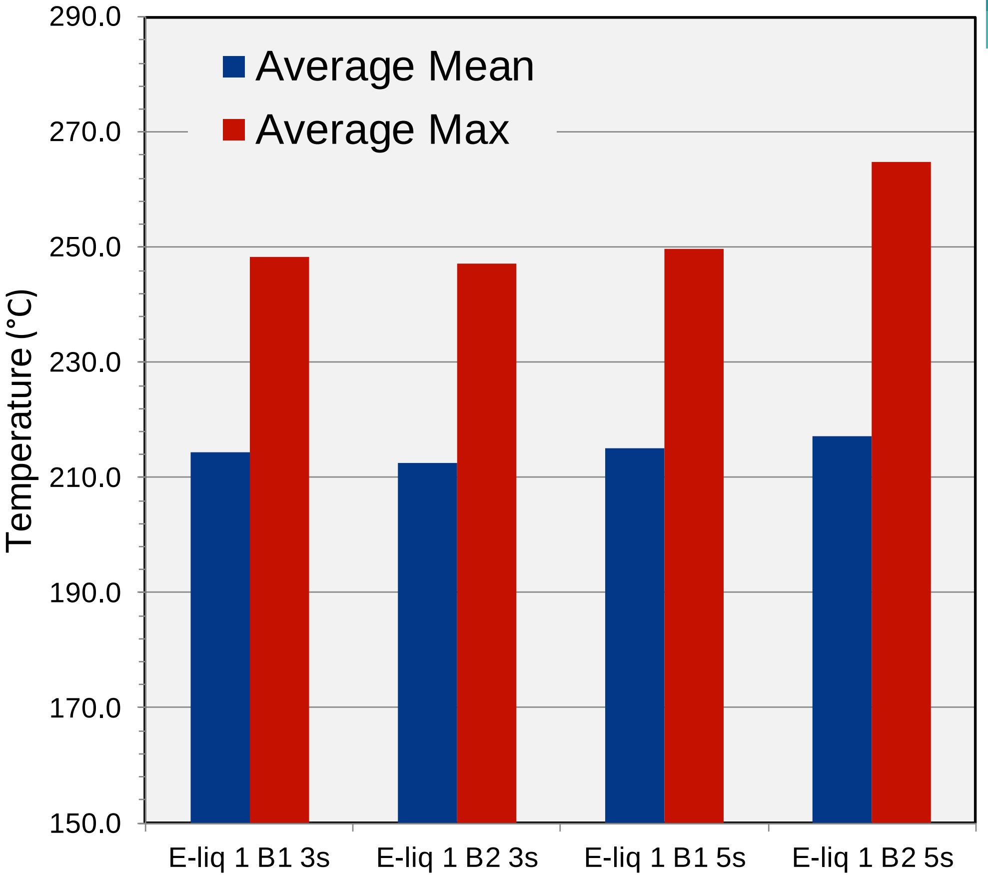
- CORESTA Puff Profile*:
  - 55cc Puff Volume*
  - 3s* and 5s Puff Durations
  - Square Wave Puff Profile*
  - 30s Puff Interval*
  - Positive pressure
- Max = Peak temperature attained in the heater zone at the hottest point during the puff (hottest spot of heater zone)
- Mean = Average temperature attained in the heater zone at the hottest point during the puff
- FLIR® factory default calibration range is 150-350°C
- We employ a custom calibration with a neutral density filter to extend the temperature range (180-464°C)
  - Segmentation is used to only include the heater zone in mean temperature calculations, values >181°C

*CORESTA Recommended Method N° 81, Routine Analytical Machine for E-cigarette Aerosol Generation and Collection – Definitions and Standard Conditions, June 2015.

Summary Averaged Temps – Single Cartridge thru One Charged Battery

- Same e-liquid, N=3
- Single battery for each replicate, 140 puffs
- Puff duration: 3s and 5s
- Prototype batteries:
  - B1 (no temperature cutoff)
  - B2 (with temperature cutoff)

- Both batteries are far below carbonyl forming temps, 350-400°C
  - Mean temperatures 212-217°C
  - Max temperatures 247-265°C



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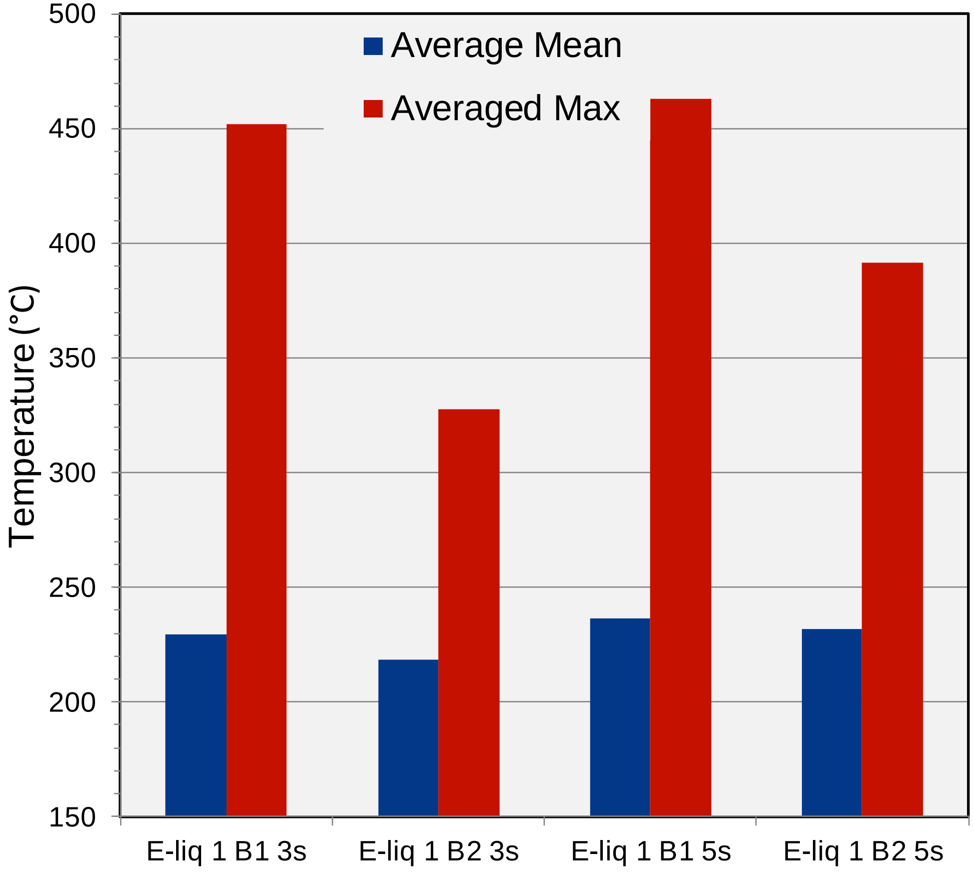
Summary Averaged Temps, Single Cartridge thru Two Fully Charged Batteries

- Simulating dry wicking
- Same e-liquid, N=3
- Two batteries for each replicate
  - First Battery (puffs 1-140)
  - Second battery (puffs 141-220)

- B1 batteries indicate max temps >350°C
- B2 with 3s puff duration <350°C
- B2 with 5s puff exceeds 350°C briefly
  - How long and how much?



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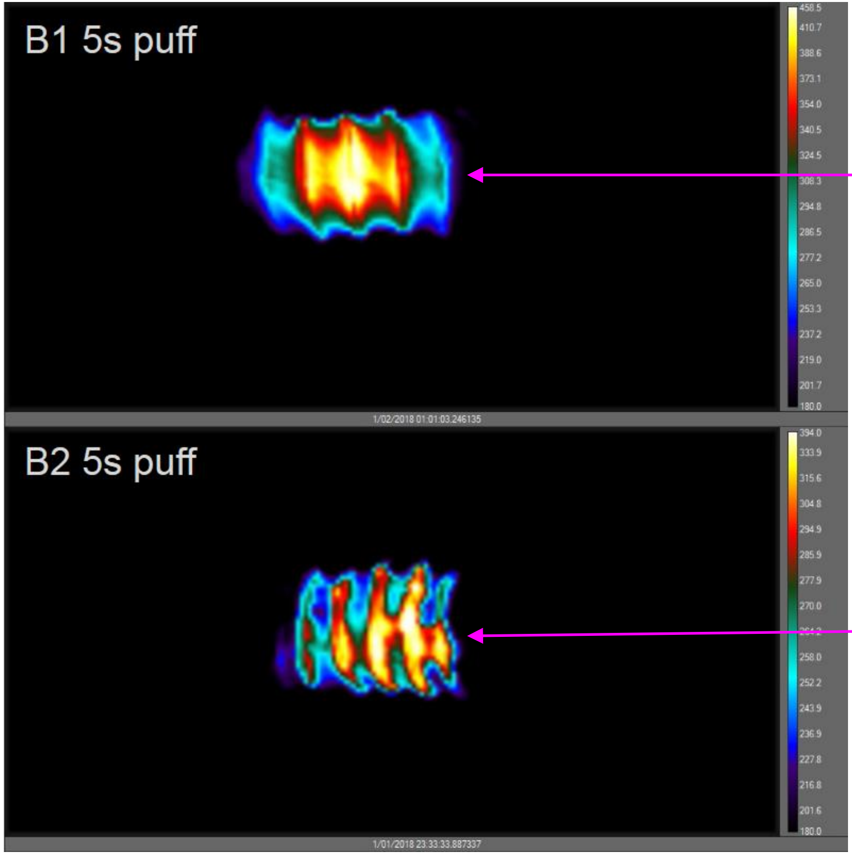
Thermal Imaging Video Capture–B1 and B2 Batteries

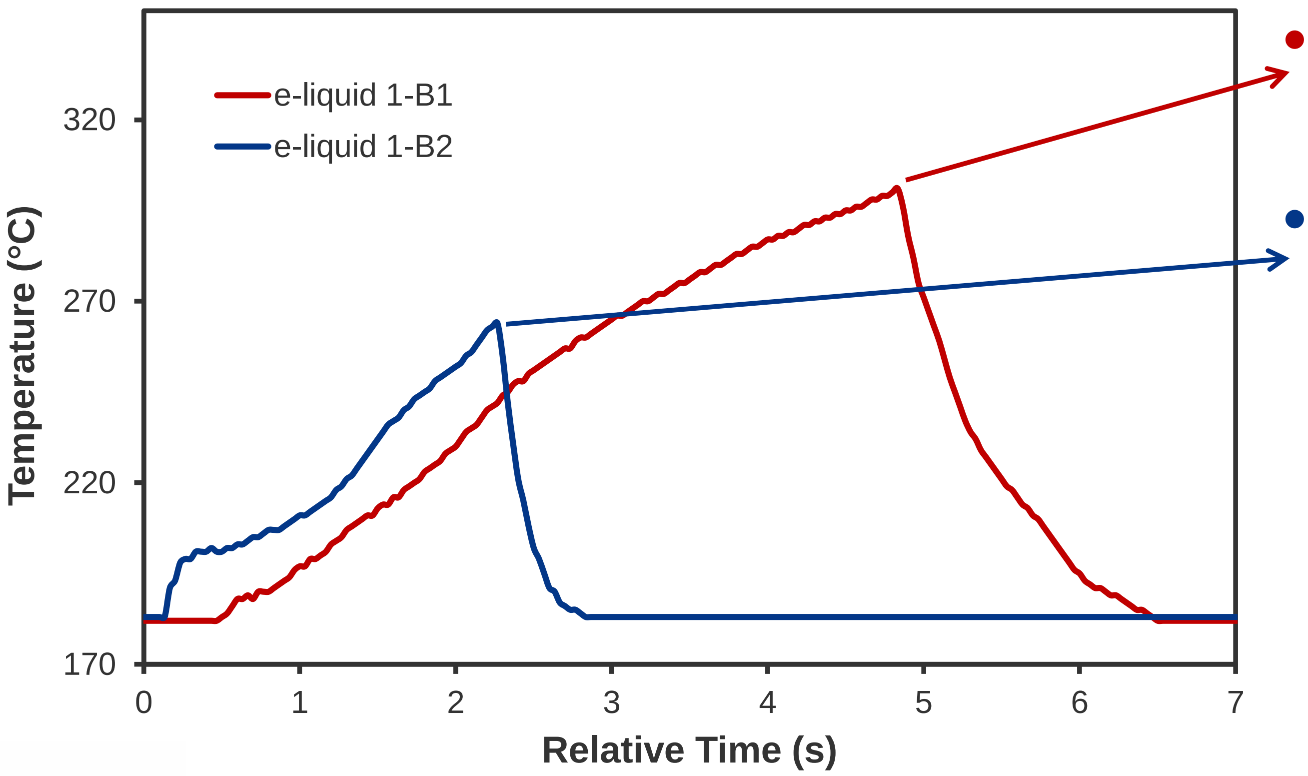
- Hottest point of puff 160 for each battery
    - 5s puff duration



No cutoff B1 battery, captured image near the end of the 5s puff

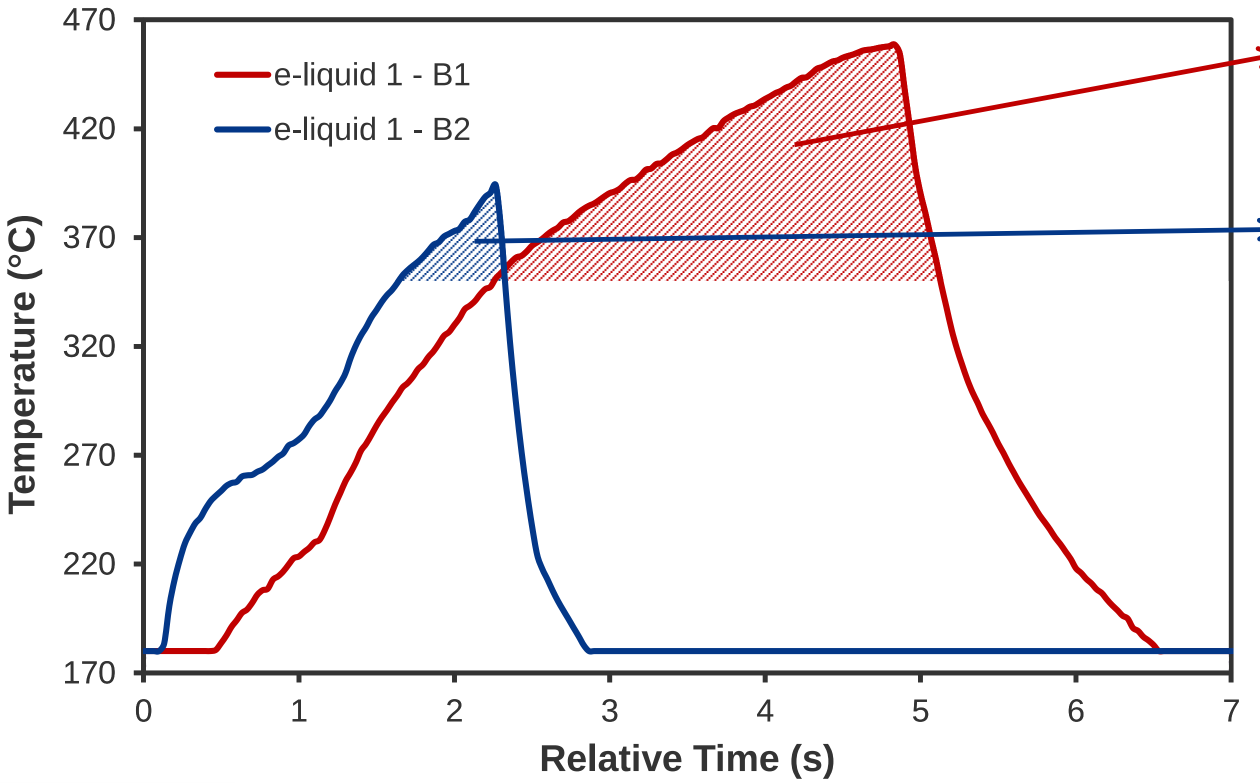
Temp cutoff B2 battery, captured image prior to cutoff (~2.2s)

E-liquid 1 Mean Temperature vs. Time, Puff 160



- B1 continues to increase in temperature thru the full 5s of puff
- Puff profiles indicate B2 battery cutoff at 2.2s into a 5s puff

E-liquid 1 Max Temperature vs. Time, Puff 160



B1 exceeds 350˚ C for 2.8s during the 5s puff ~ 60%

B2 exceeds 350˚ C for 0.63s ~ 13%

Further analysis of the data can show the area (pixel) percentage above 350˚ C

B1 ~ 27% of pixels >350˚ C
B2 ~ 2% of pixels >350˚ C

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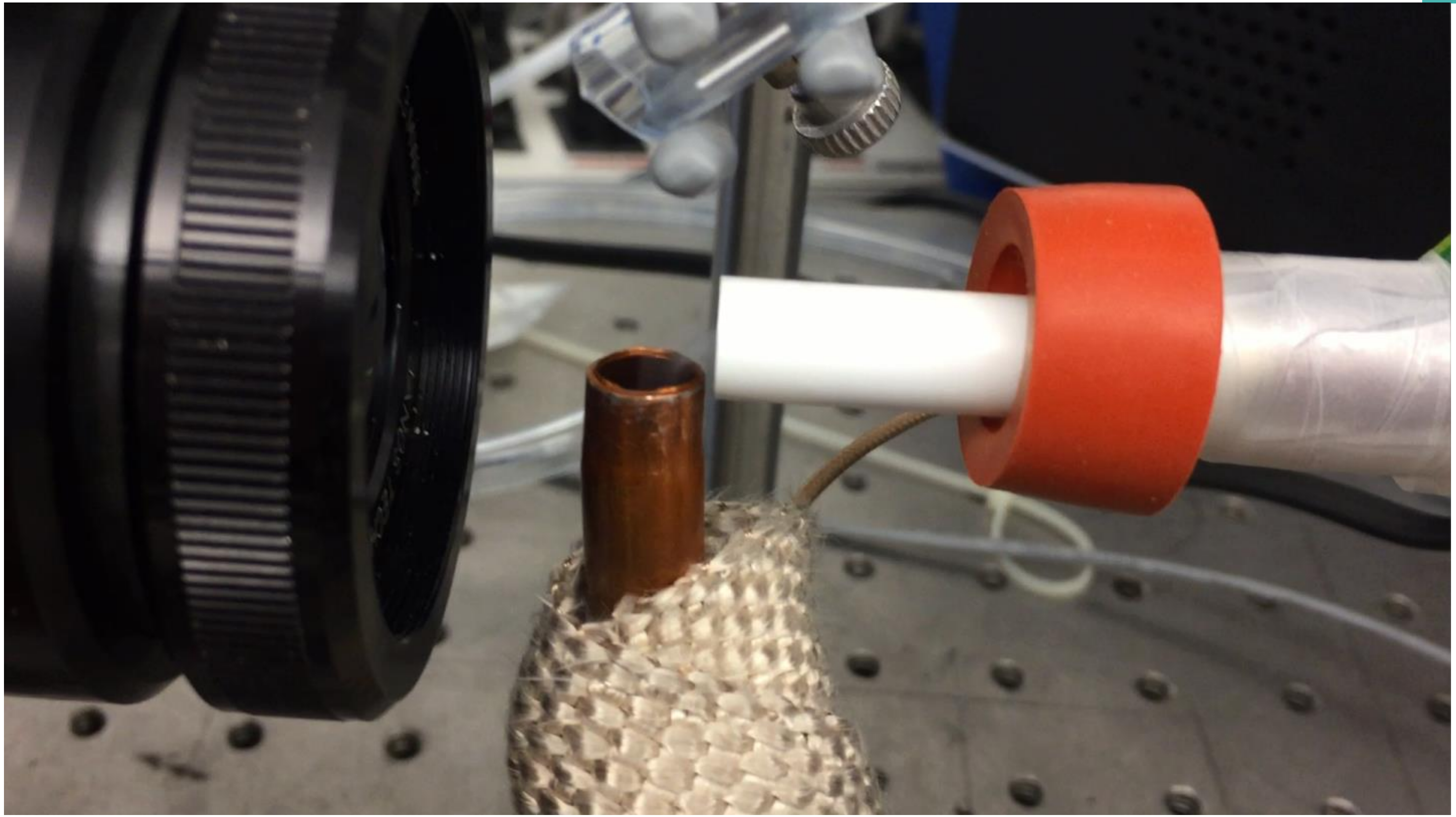
Simultaneous FLIR® & FTIR Formaldehyde Analysis

Vapors produced during each puff are simultaneously measured by FLIR thermal imaging and FTIR for formaldehyde concentration

- Vapors drawn into FTIR using temperature controlled tubing



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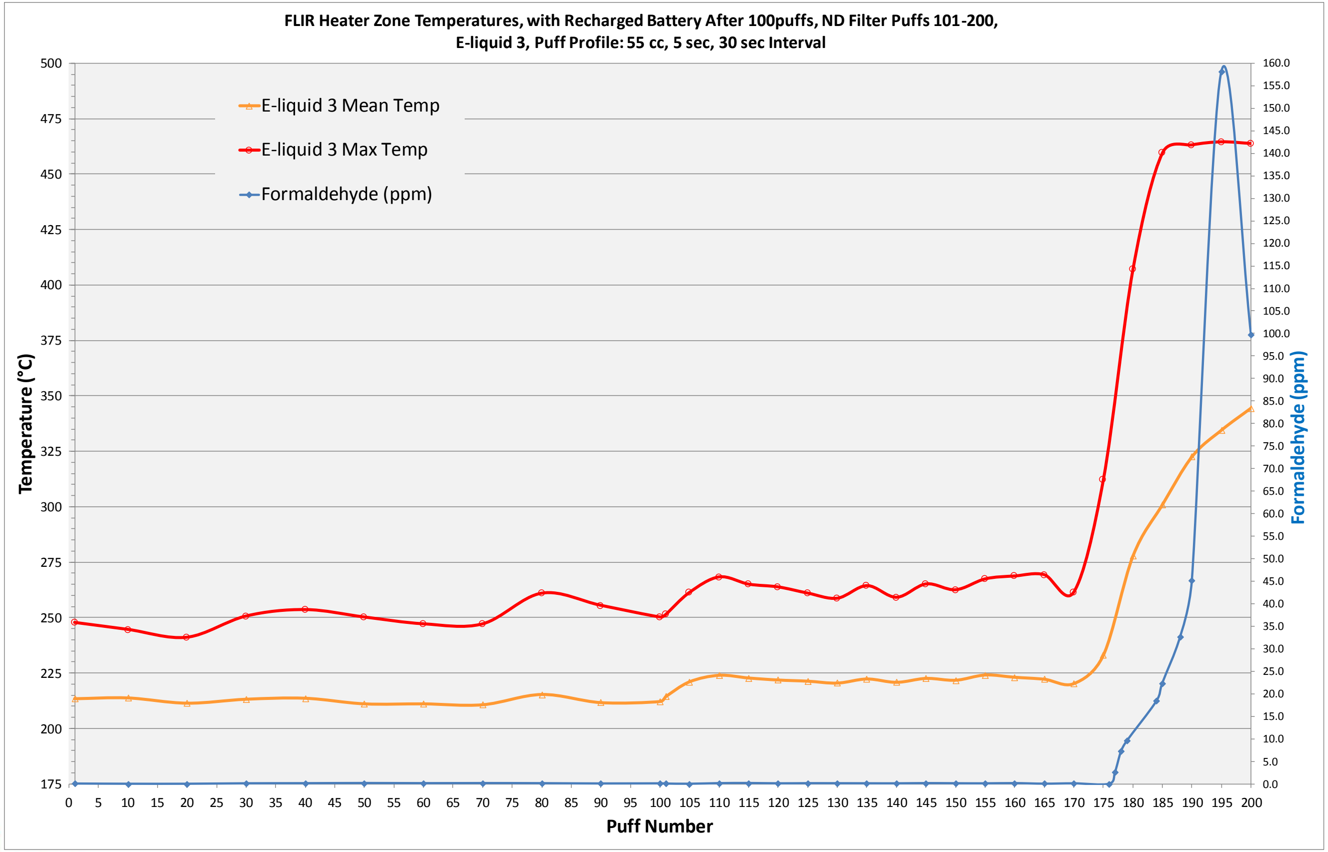
FLIR Heater Zone Temp w/ Real-Time FTIR Formaldehyde



In this example, Formaldehyde begins to evolve between 350-400°C, using B1 battery.

Conclusions

- FLIR® imaging overcomes drawbacks of thermocouple heater core measurements of ENDS devices.
- FLIR ® imaging provides real time puff by puff temperature data, which can be automated.
- Emissivity and other parameters can be determined using ASTM methods and confirmed using thermocouple measurements.
- FLIR ® measurements of the heater zone mean and max temperatures from the hottest frame during a puff provides consistent comparison of different e-liquids and ENDS devices.
- FLIR ® imaging and FTIR gas analysis can be simultaneously used to provide real-time formaldehyde measurements and heater core temperatures.

Acknowledgments

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