

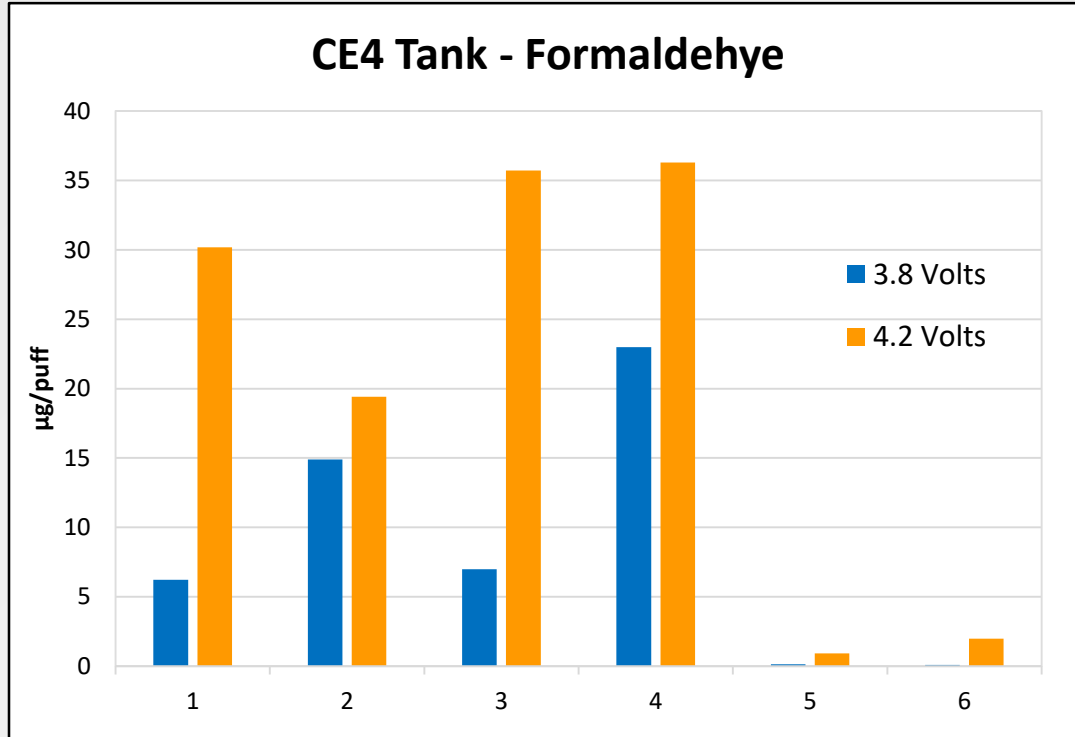
Characterization of a Temperature Regulated Electronic Nicotine Delivery System for Potential use as a Reference Device

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Why do we need a reference device?

- Method Development & Validation
 - Reference device
- Collaborative Study samples
 - r & R values
- Process monitor control
 - Routine sample analysis

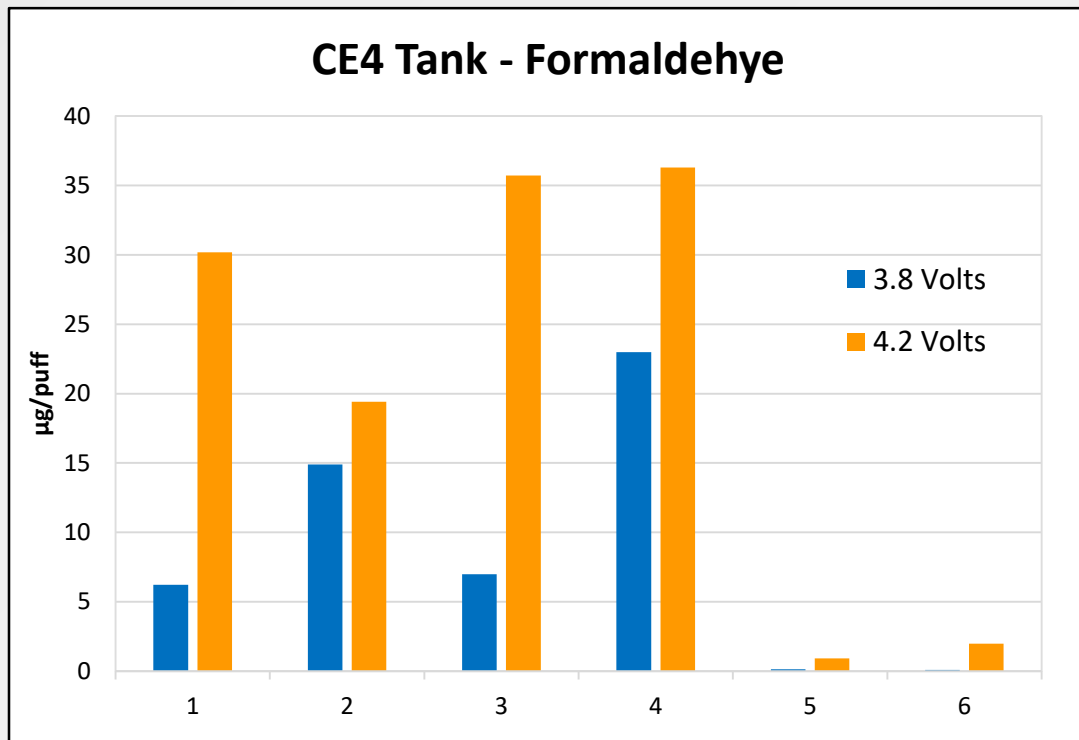
Variability in an Issue



Data from CE4 tank based devices. Three unique coils with two replicates collected per coil.

Samples were collected at two battery voltages.

Variability is an Issue



CE4	Formaldehyde µg/puff	Formaldehyde µg/puff
Rep	3.8 Volts	4.2 Volts
1	6.21	30.18
2	14.89	19.42
3	6.97	35.72
4	22.99	36.30
5	0.13	0.91
6	0.08	1.97
AVG	8.55	20.75
SD	8.9	16.1
% RSD	104.5	77.8

Evidence of Coil Overheating



Unused coil



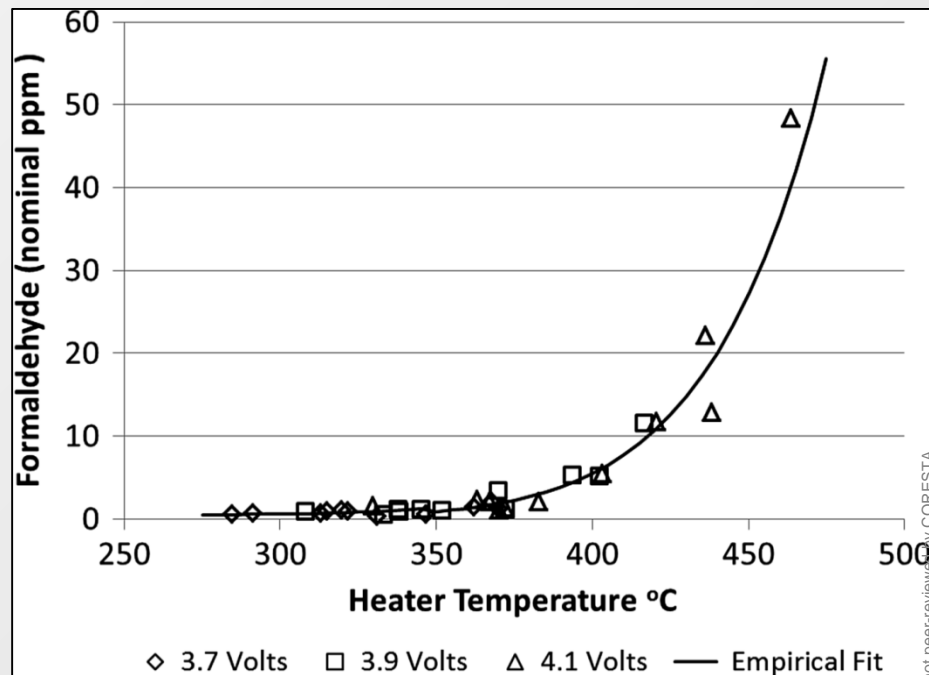
Used coil

Evidence of charring on wick
(example from CE4 tank)

Aldehyde Temperature Dependence

During heating of the e-liquid, thermal degradation leads to the formation of formaldehyde, acetaldehyde, and acrolein

The formation of aldehydes, as a function of coil temperature, has been found to have an exponential relationship, leading to higher production of aldehydes at high temperatures



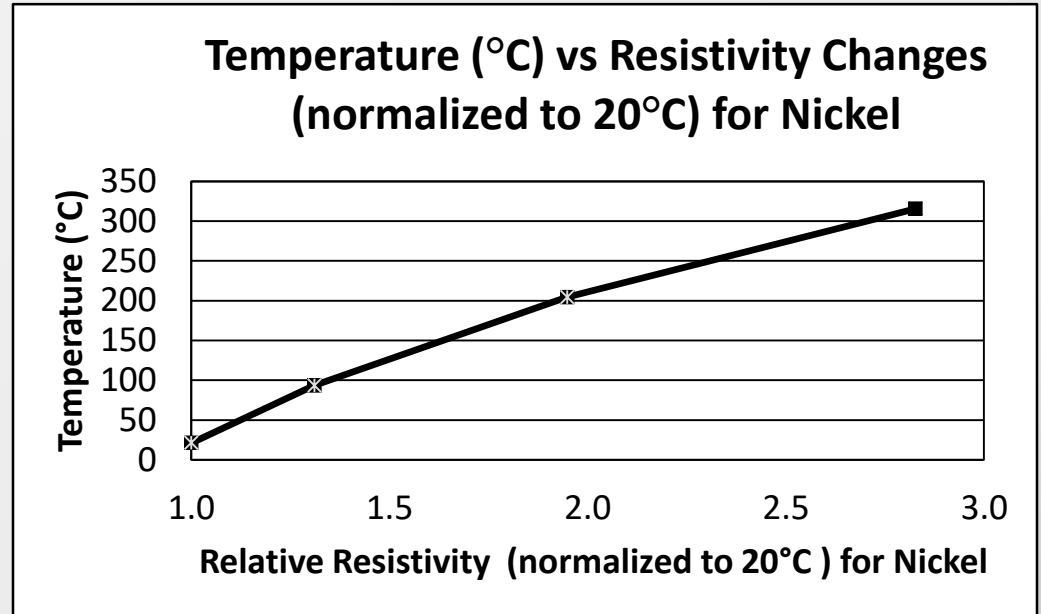
Data from: Flora et al. 2017

Temperature Regulated (TR) Devices

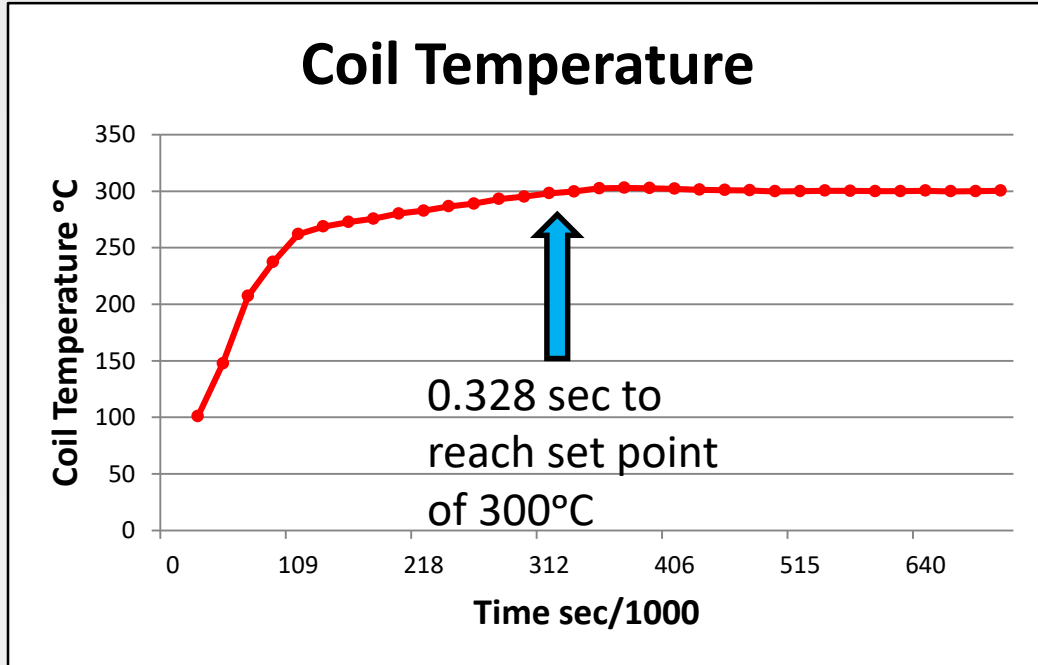
- A possible solution to device variability
 - Eliminate coil overheating
 - Occurs when either too much power or not enough liquid is supplied to the atomizer
 - Uniform and consistent delivery aldehydes
 - Known delivery of aldehydes at a given coil temperature

Temperature Regulated Devices

The relationship of the relative resistivity of the coil material to the temperature of the coil allows for accurate monitoring and control of the vaping temperature



Temperature Profile (Pre-Heat off)



Temperature rises rapidly to the set temperature (300 C) and maintains set temperature over the full activation time of the device

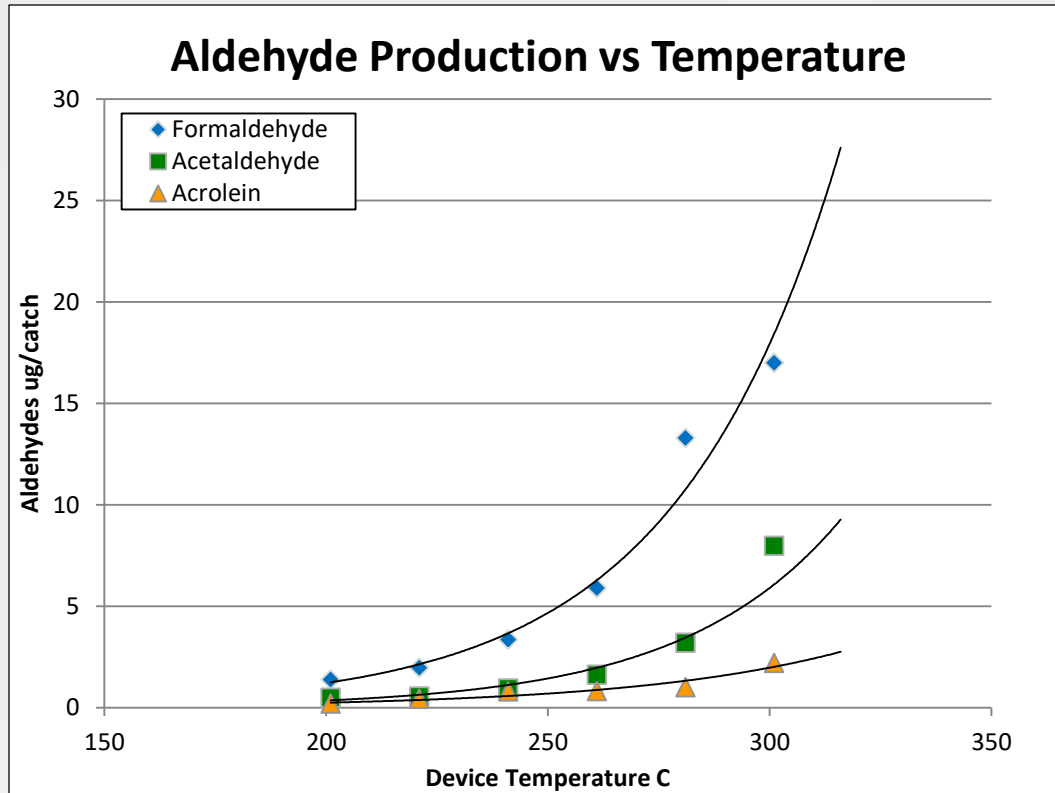
Device Characterization

- How much E-liquid does the device consume?
- What concentration of aldehydes is produced per gram of e-liquid consumed?
- Does the device produce the same amount of Aldehydes each time?

Analytical Methodology

- Aerosol was collected using an automatic “button pusher” using a 55mL constant flow puff over 4 seconds every 30 seconds
- E-liq: 50:50 PG/VG with 2% nicotine was used with all samples
- Samples were collected using new coils for each device, with the device pre-heat function off
- Aerosol samples were analyzed for aldehydes (formaldehyde, acetaldehyde and acrolein).

Yield Profiles vs Temperature



Yield profile from a single device was generated to determine optimal conditions for aldehyde production

Data was collected from 200-300 C

The device showed an exponential relationship of aldehyde production versus temperature

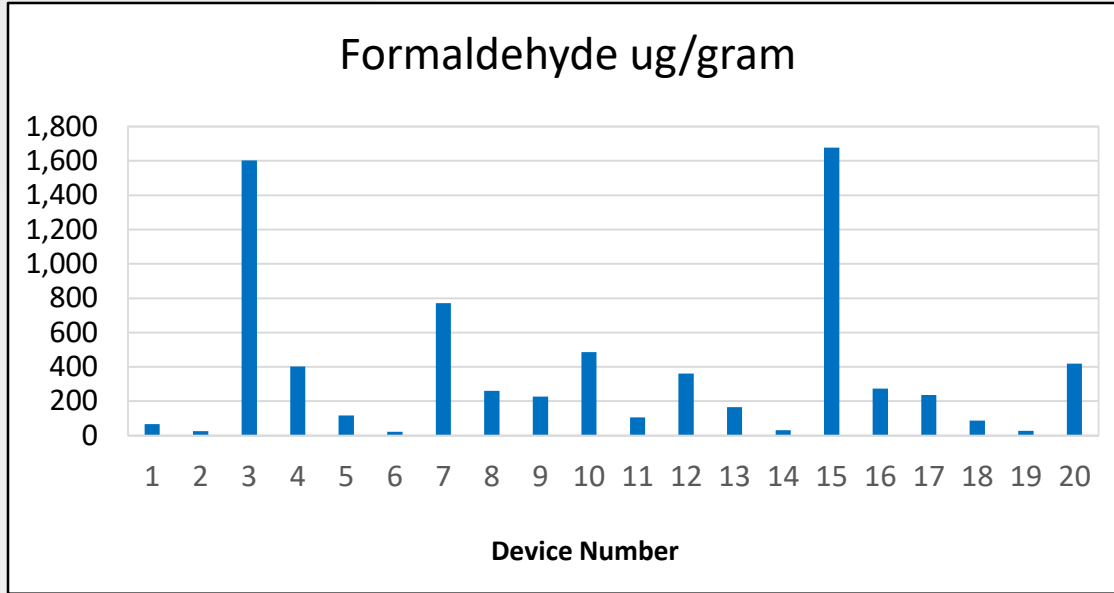
Yield Profiles vs Temperature

	Temperature °C	Formaldehyde µg/sample	Acetaldehyde µg/sample	Acrolein µg/sample
Band of relatively low-level aldehyde production	201	1.38	0.49	0.20
	221	1.97	0.56	0.44
	241	3.36	0.94	0.80
	261	5.83	1.62	0.82
Band of analytically relevant aldehyde production	281	13.3	3.20	1.01
	301	17.0	7.98	2.21

Experimental Design

- Native delivery at higher temperature
 - Across device variability
 - With-in device variability
- Fortified E-liquid delivery at a lower temperature
 - E-liquid spiked with formaldehyde and acetaldehyde

Across Device Variability



- Data collected from 20 devices under identical conditions
- Devices 2,6,14,19: Yielded approximately same levels of formaldehyde production
- Overall: Based on the RSD of the 20 devices, TR devices do not deliver consistent levels.

	Weight Loss mg/puff block	Acetaldehyde ug	Acrolein ug	Formaldehyde ug
RSD	33.1%	200.4%	263.2%	129.1%

Within Device Variability

Device	Acetaldehyde		Acrolein		Formaldehyde	
	µg/g	% RSD	µg/g	% RSD	µg/g	% RSD
1	64.92	21.8%	8.97	4.8%	269.76	8.2%
2	40.29	29.9%	6.54	8.2%	141.05	16.7%
3	58.16	26.1%	7.58	10.7%	158.60	33.3%
4	90.20	62.3%	9.64	94.3%	258.35	73.0%
5	39.23	18.9%	4.15	17.2%	115.87	7.8%
6	4.43	6.0%	3.59	10.4%	25.41	30.2%

6 devices (same coil) with 6 replicates per device were collected.

Half of these devices gave a reasonable %RSD (<20%)

Complete characterization of the device and coil is necessary for use as a reference product

Transfer of Fortified E-liquids

- Reference liquid spiked with formaldehyde and acetaldehyde
 - Samples were collected at 231 C
 - Data collected from five devices and batteries
 - Data presented on a per gram basis
 - This approach has been used in our laboratory to validate trapping capacity and efficiency for new methods

Spiked Reproducibility

Data from the spiked E-liquid collected on the TR devices.

Sample	Yield mg/puff block
1	136
2	281
3	323
4	116
5	266
Average	224
RSD	41.3%

Recoveries of Aldehyde were $\pm 20\%$ of their target value

Results Summary

- Inconsistent production of aldehydes was found in the 20 devices tested, with across device variability of 100%-200%.
- 50% of the devices gave acceptable, with-in device variability, of <20% (n=6).
- Uniform delivery of aldehydes was obtained using a fortified liquid, variability of <10%.

Conclusion

- Temperature regulated devices are not suitable as a reference product for producing analytical levels of native aldehydes
- With prior characterization a single tank, battery and coil may function as a reasonable in laboratory reference product
- Temperature regulated devices were found to be suitable for use as a reference product when using a low coil temperatures to transfer an e-liquid of known aldehyde concentration

Thank you for your attention