Juul Labs Science

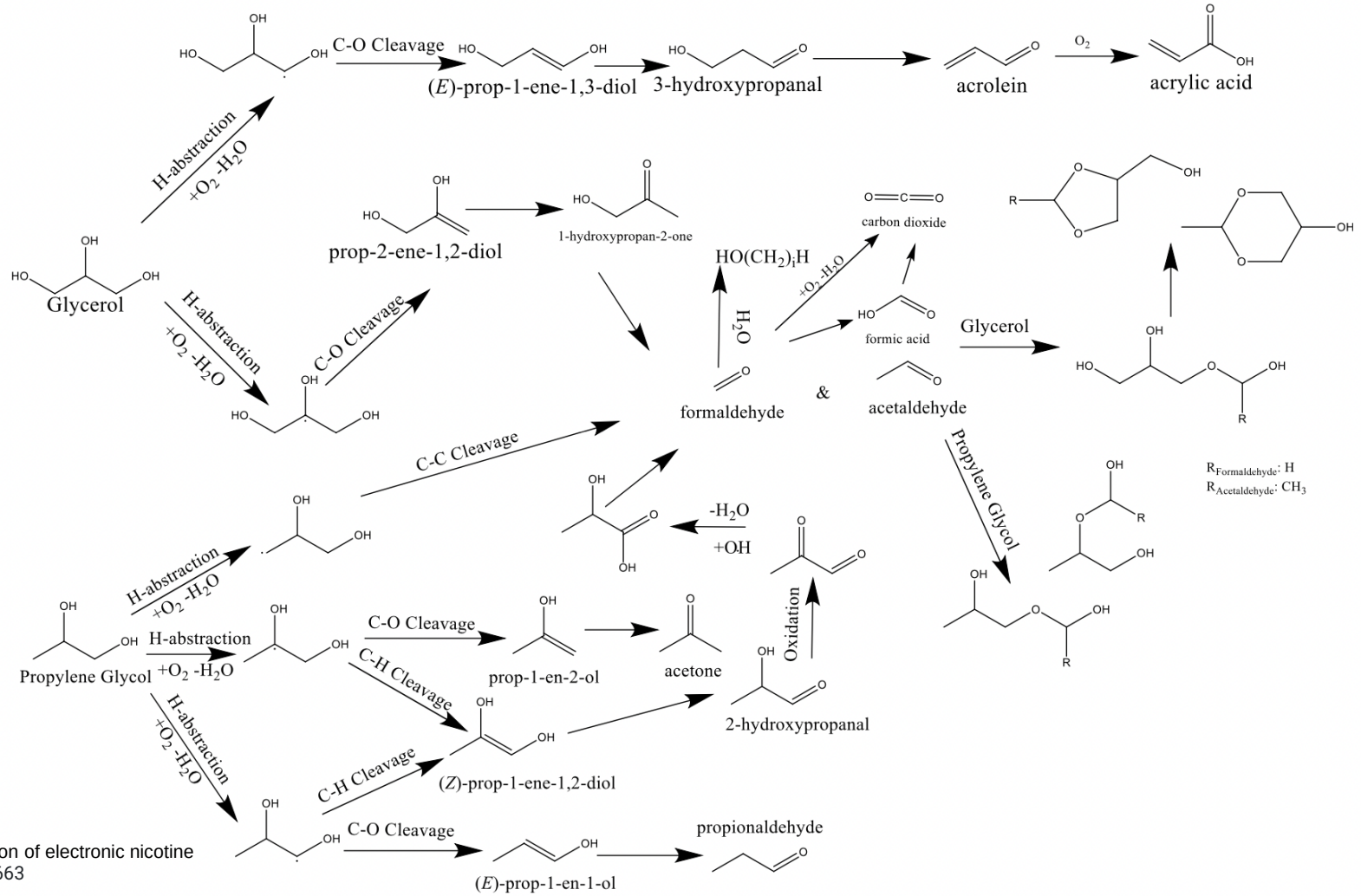
Assessment of Formaldehyde and Acetaldehyde Formation in E-Liquid and During Puffing of an ENDS Product

Manali Aggrawal , Klavdija Bukovec, Jason Chan, Bob Moision, Kate Pearce

76th Tobacco Science Research Conference
September 2023

Aldehyde Formation in E-Liquid Carrier Liquids

- Chemical pathway for low temperature aldehyde formation from liquid phase ENDS liquids [1]
- Aldehyde formation during stability testing can be used as an indicator of formulation instability



[1] Jaegers, N.R., Hu, W., Weber, T.J., Hu, J.Z.; Low-temperature (< 200 °C) degradation of electronic nicotine delivery system liquids generates toxic aldehydes; 2021 ♦ *Scientific Reports* ♦ ID# 11663

Goals

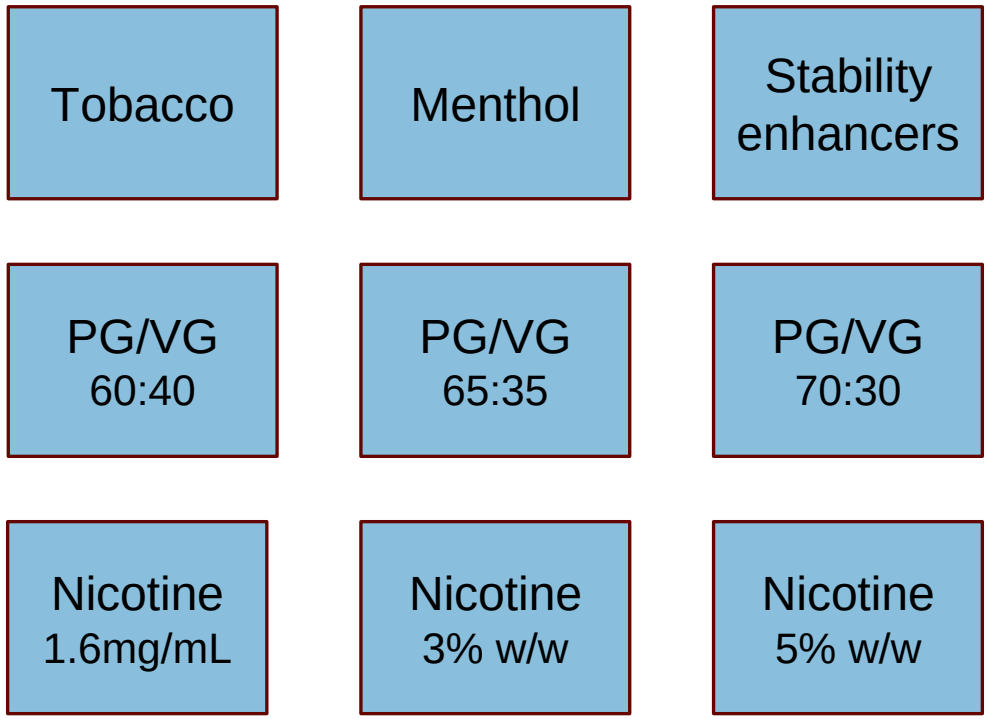
- Understand the role of formaldehyde and acetaldehyde formation in e-liquid pods *vs.* through thermal degradation during aerosolization

Aldehydes form during aerosolization of the e-liquid through thermal degradation pathways of e-liquid components

Aldehydes form in the e-liquid during pod storage, under standard (25˚C/60%) RH and accelerated (40˚C/75% RH) conditions

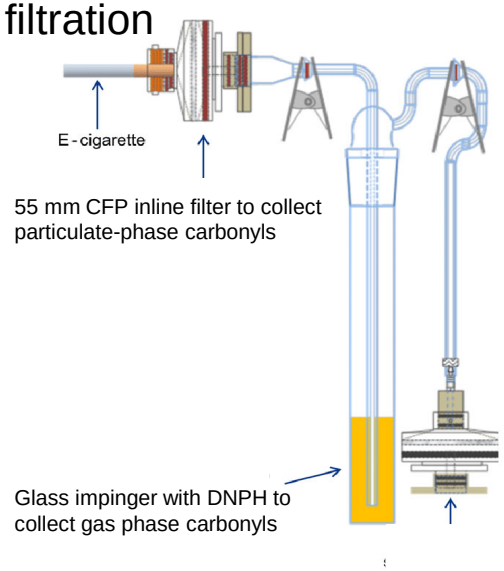
E-Liquid Study Design

- Filled pods were evaluated freshly made (T0) and after 30 days aging at 40˚C/75% RH (T30)
- A total of 27 formulations were tested
- E-Liquids chosen had demonstrated a range of stability from total condensate testing



Sample Collection and Analysis

- Total Condensate collection of aldehydes on CFP and in DNPH-filled impingers followed by extraction and filtration



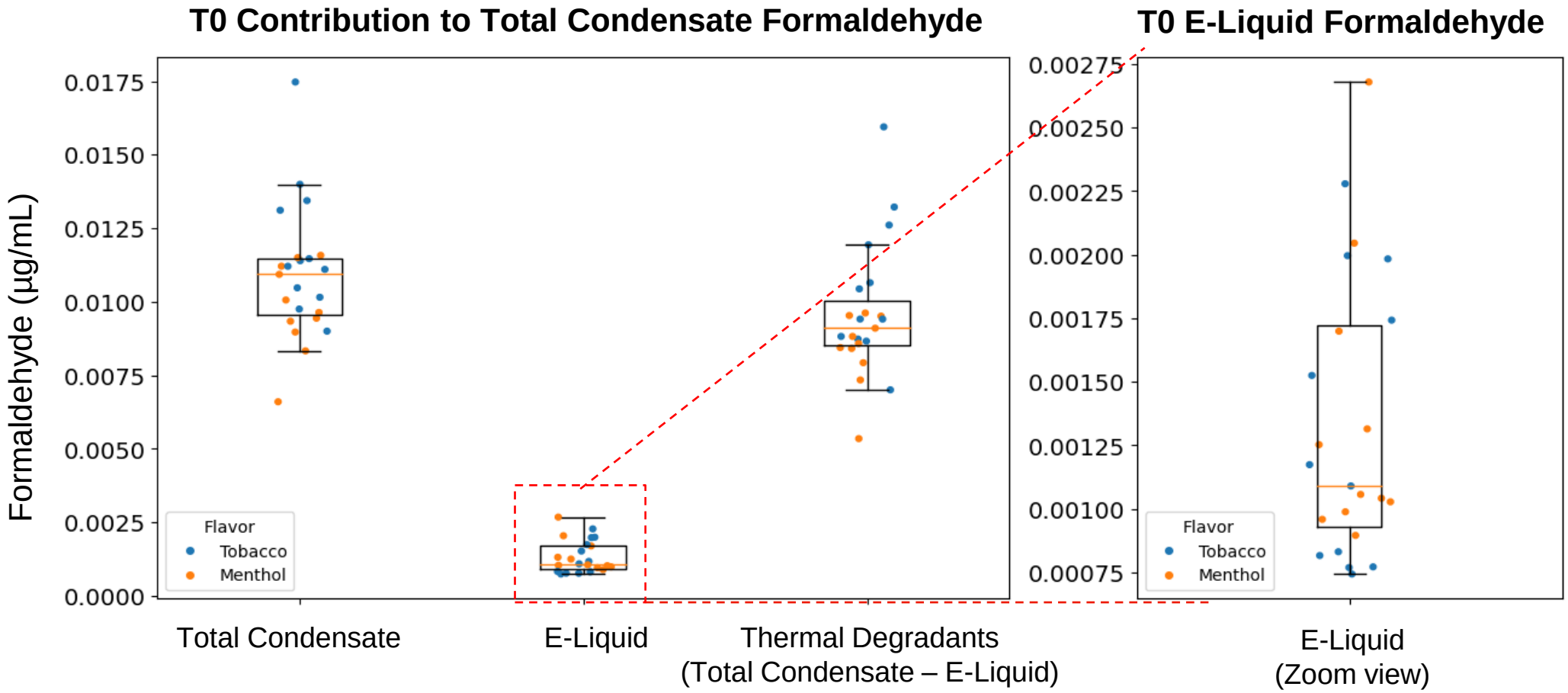
- Puffing Regime
  - Puff Volume: 110 mL
  - Puff duration : 6 sec
  - Puff Interval : 30 sec
  - Puff profile: Square,
  - Puffs per collection: 50

- E-Liquid samples extracted from the pod, added directly to DNPH solution

- Analyzed via
  - Agilent 1290 HPLC-MSD
  - Waters Acquity BEH C18 2.1mm x 50mm, 1.7um particle
  - Agilent Chemstation
  - 3 replicates per sample, 3 injections per replicate

- Method based on CORESTA RM 74

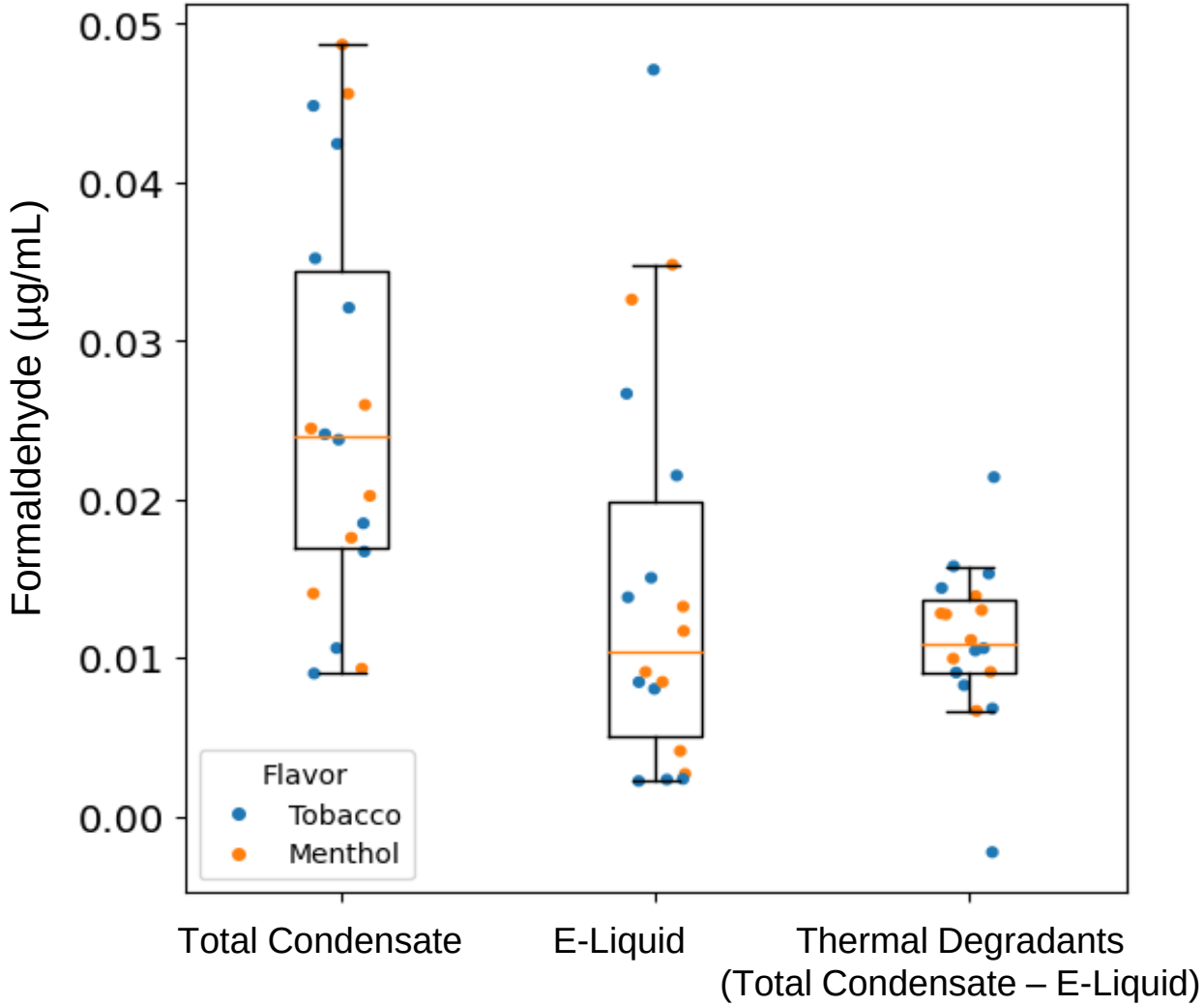
Formaldehyde levels at T0



- Major contributor: thermal degradation formaldehyde
- Minor contributor: e-liquid formaldehyde (10x smaller)
- Tightly clustered sample set

Formaldehyde levels at T30

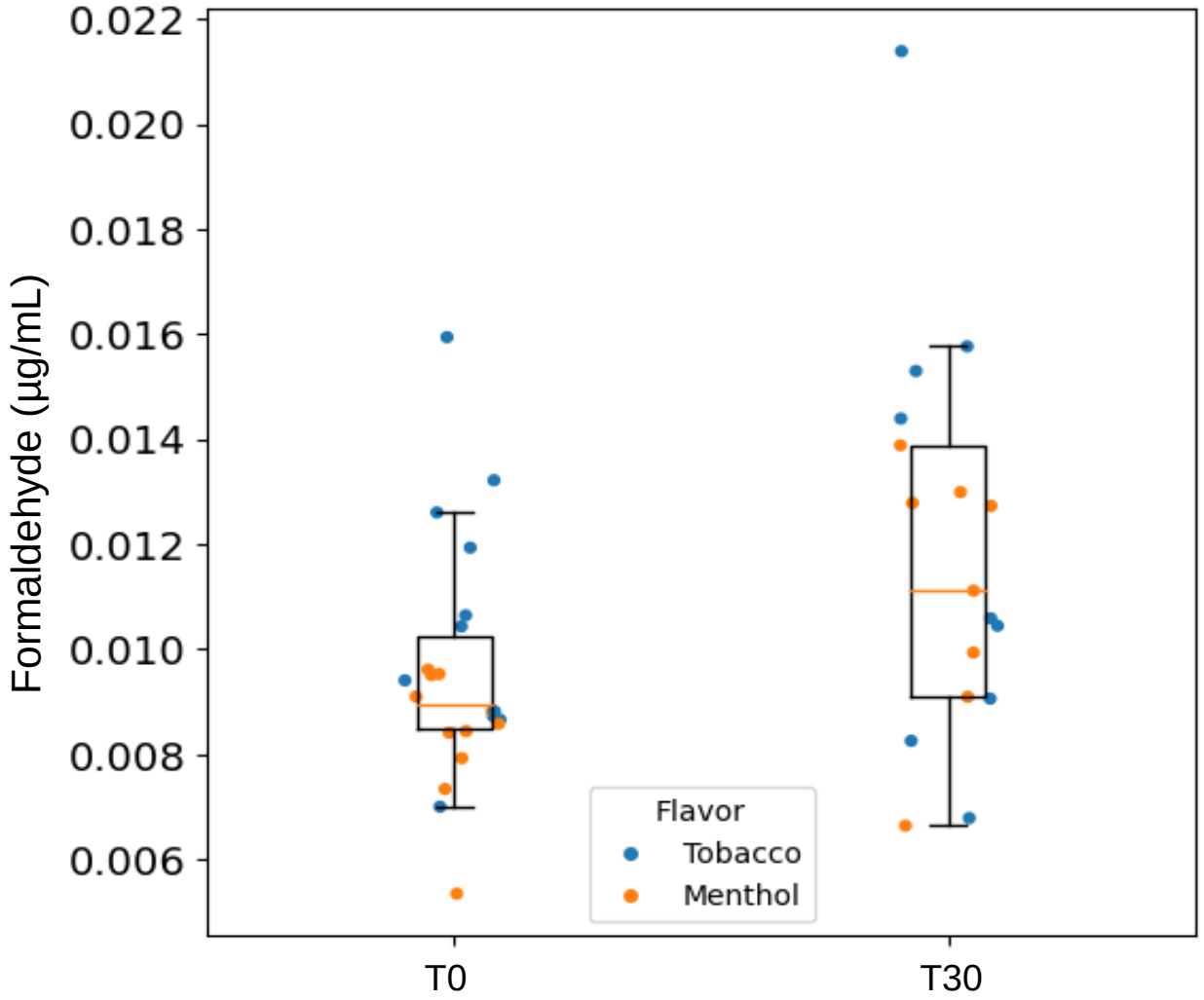
T30 Contribution to Total Condensate Formaldehyde



- E-Liquid and thermal degradant formaldehydes equivalent contributors
- E-Liquid formaldehydes contribute significant spread driven by formulations with varying stability

Thermal Degradation Formaldehyde at T0 and T30

Comparison of T0 and T30 Thermal Degradant Formaldehyde

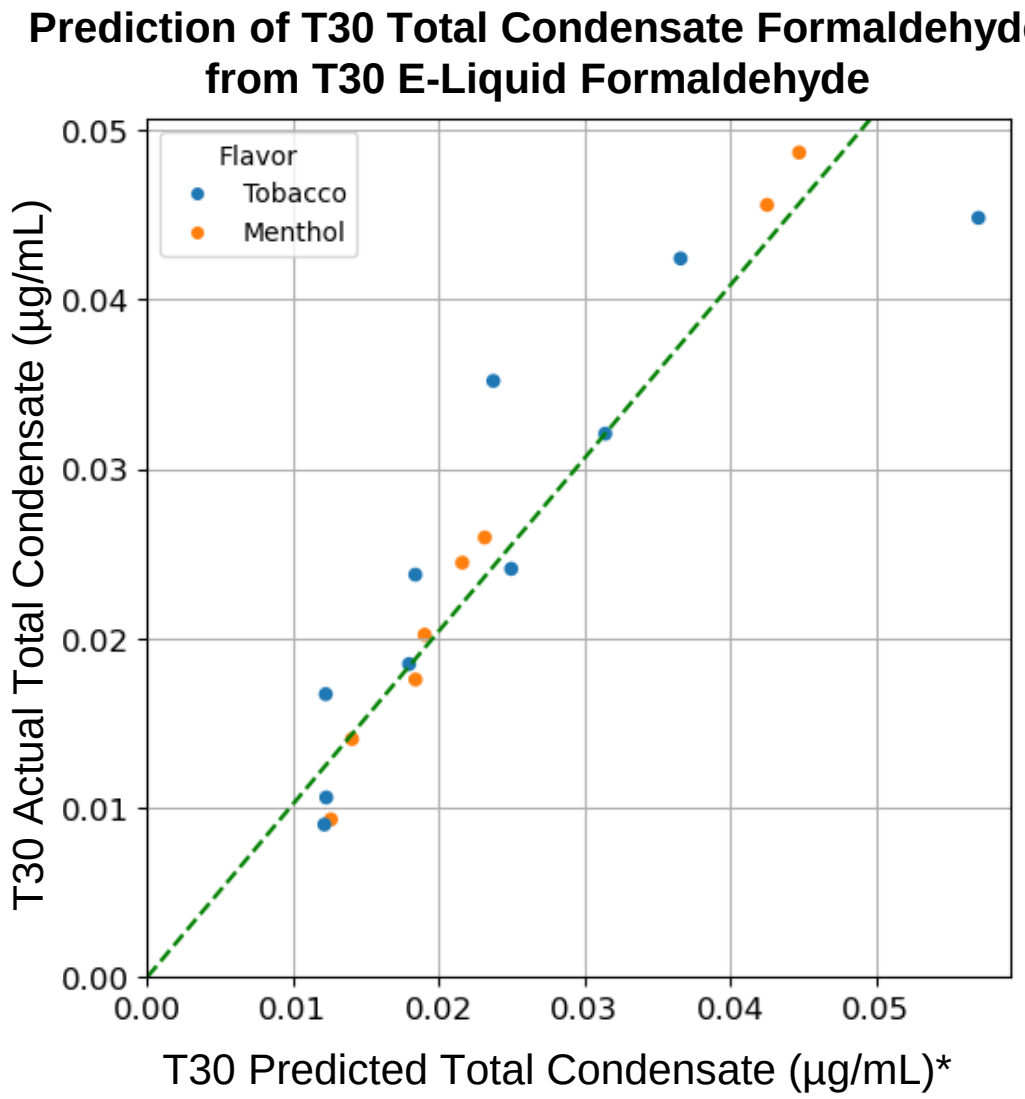


- Thermal degradant formaldehyde values approximately constant regardless of the formulation stability

- Can we use average Thermal Degradation formaldehyde value to predict Total Condensate formaldehyde from measured E-Liquid formaldehyde?

E-Liquid Formaldehyde as a Screening Method for Stability

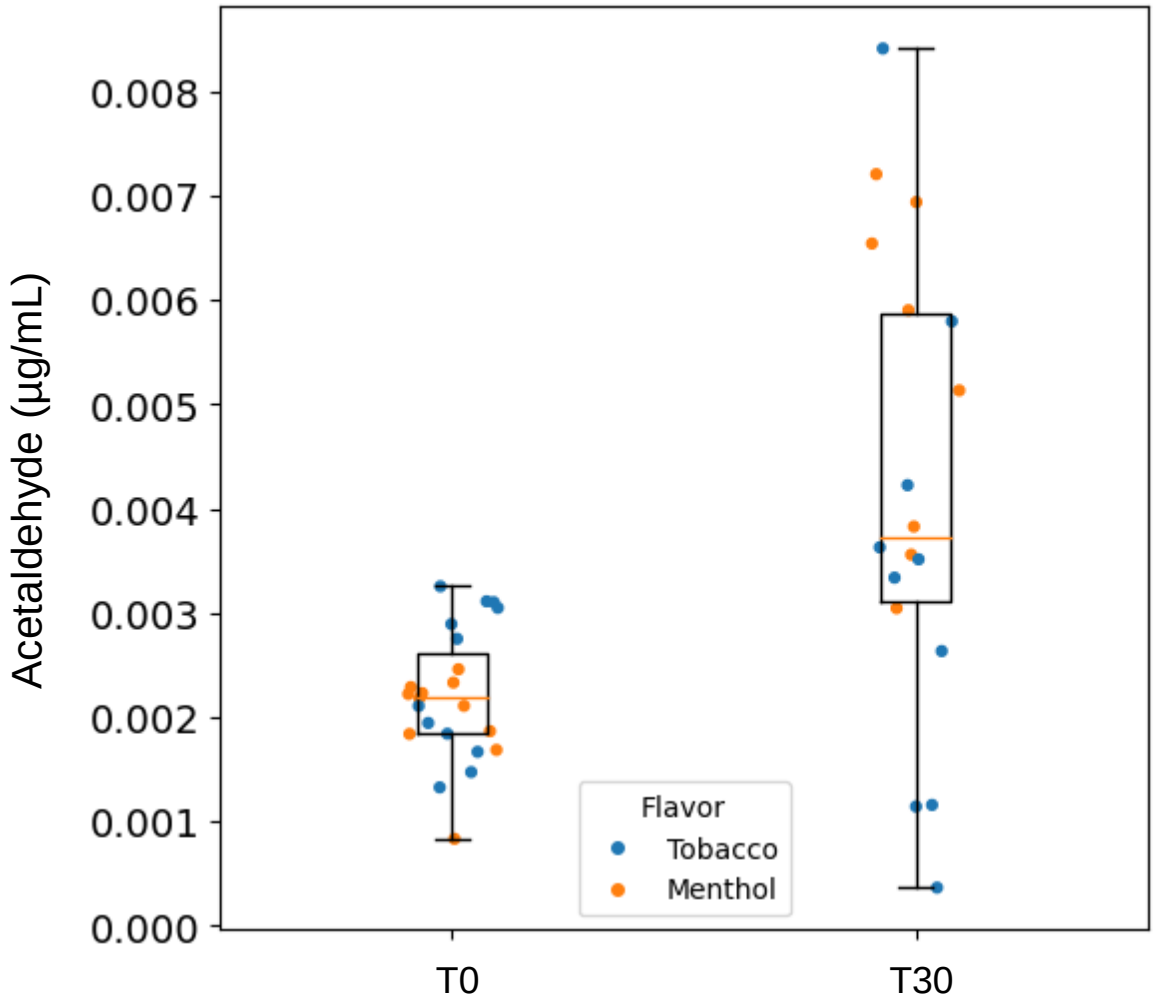
- Data shows
  - Slope = 1.01 for actual vs predicted total condensate
  - Using average Thermal Degradation formaldehyde value to predict Total Condensate from measured E-Liquid formaldehyde is a good approximation



* T30 Predicted Total Condensate =
T30 E-Liquid +
T0 Average Thermal Degradant (across all formulations studied)

Thermal Degradation Acetaldehyde at T0 and T30

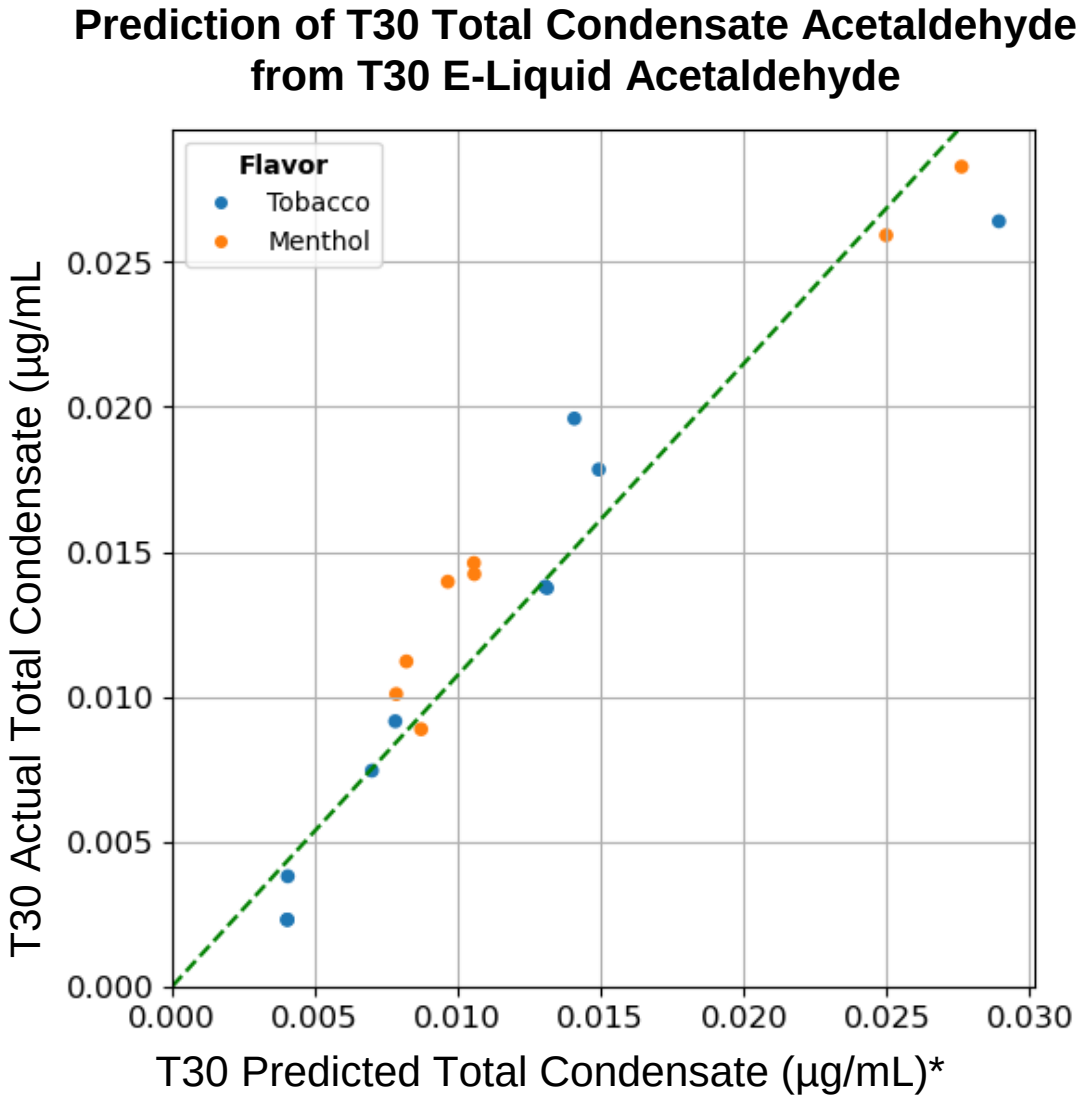
Comparison of T0 and T30 Thermal Degradant Acetaldehyde



- Thermal degradant acetaldehyde values behave differently
- At T0, thermal degradant value is small and tightly clustered
- At T30, thermal degradant values have increased with sigificant data spread

E-Liquid Acetaldehyde as a Screening Method for Stability

- Data shows
  - Slope = 1.16 for actual vs predicted total condensate
  - Predicted Total Condensate Acetaldehyde levels overestimate the measured values by ~16% for this sample set

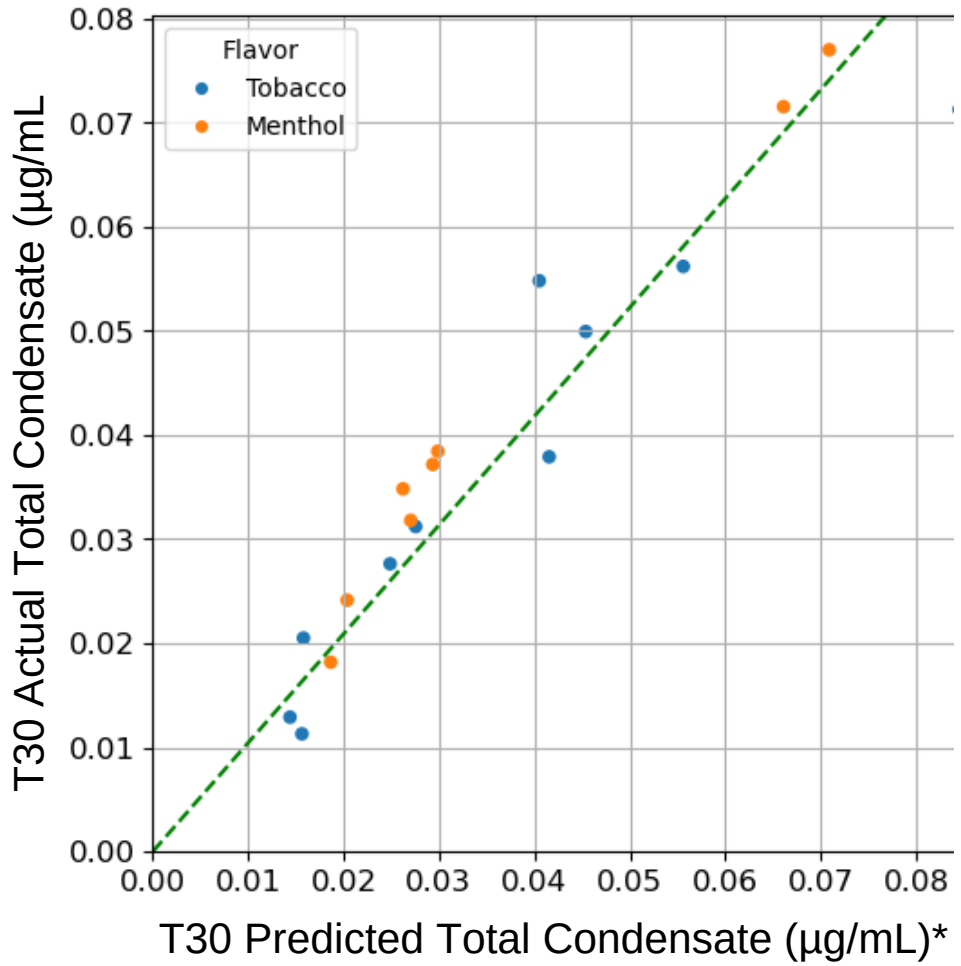


* T30 Predicted Total Condensate =
T30 E-Liquid +
T0 Average Thermal Degradant (across all formulations studied)

Combined E-Liquid Formaldehyde and Acetaldehyde Levels

- Data Shows
- Slope = 1.05 for actual vs predicted total condensate
- Total Condensate (Formaldehyde + Acetaldehyde) levels overestimate the measured values by ~5% for this sample set
- Using average Thermal Degradation formaldehyde and acetaldehyde values to predict Total Condensate from measured E-Liquid formaldehyde and acetaldehyde values is a fair approximation

Prediction of T30 Total Condensate Formaldehyde + Acetaldehyde from T30 E-Liquid Formaldehyde + Acetaldehyde



* T30 Predicted Total Condensate =
T30 E-Liquid +
T0 Average Thermal Degradant (across all formulations studied)

Conclusions

- Formaldehyde and acetaldehyde are formed both in the e-liquid pod during aging and as a result of thermal degradation due to aerosolization
- Formaldehyde and acetaldehyde formation as a result of formulation instability during pod aging happens mostly in the e-liquid and not due to thermal degradation during aerosolization
- Formaldehyde (and to a lesser extent acetaldehyde) formed due to thermal degradation during aerosolization is approximately constant (for the sample set studied) and aging time (for 40˚C/75% RH storage for 30 days)
- Possible to use e-liquid formaldehyde and acetaldehyde values as a rapid screening method to predict relative formulation stability

Acknowledgments

Manali Aggrawal, Senior Scientist, Analytical Chemistry

Klavdija Bukovec, Research Associate, Analytical Chemistry

Jason Chan, Senior Research Associate, Analytical Chemistry

Bob Moision, Principal Scientist, Team Lead, Analytical Chemistry

Vincent Nip, Senior Research Associate, Analytical Chemistry

Thank you for your attention

Any Questions?