# Headspace Analysis of E-Cigarette Liquid Flavorings by Vacuum Thermal Desorption

Eric Knappenberger, Corey N. Stedwell, J. Daniel DeBord

1st Detect Corporation, Webster, TX

## Overview

Here we demonstrate analysis of E-Cigarette flavoring via headspace sampling utilizing a Vacuum Thermal Desorption (VTD) system, simple sample preparation and a standard addition method.

## Introduction

The increasing popularity of E-Cigarettes as an alternative to tobacco and the pending regulation of E-cigarettes have brought about the need for tailored chemical analyses. E-Cigarette flavoring compounds have previously been analyzed using other mass spectrometry based techniques typically have longer analysis times and sample conditioning requirements. Here we present a method for direct analysis of flavoring compounds in E-Cigarette Liquids utilizing headspace analysis with minimal sample preparation. This is achieved using a VTD system which is coupled to a miniaturized cylindrical ion trap mass spectrometer. This technique is capable of providing sensitive analysis of these volatile flavoring compounds with analysis cycle times on the order of 2-3 minutes.

## Methods

A variety of E-liquid solutions were analyzed using an MMS-1000 (1st Detect, Webster, TX) configured with a VTD concentration system. The VTD functions by drawing the sample vapor across a sorbent coated mesh at ambient temperature and pressure. Headspace analysis can be achieved by pulling this gas from a vial containing a volatile liquid sample. The low concentration analyte vapor entrained in the gas flow can be captured by the sorbent mesh, with longer sampling times resulting in lower limits of detection. The chamber containing this sorbent mesh is then evacuated, opened to the analytical vacuum chamber, and heated to cause thermal desorption and transfer of the adsorbed analytes to the mass spectrometer. Samples of E-liquid were diluted to 0.5g/mL in deionized water to reduce matrix effects and standard addition methods were utilized to evaluate the concentration of specific flavoring compounds. The quantification was performed by integrating desorption peaks for characteristic analyte ions. The E-Cigarette flavors 'Lemon', 'Vanilla' and 'Cinnamon' were analyzed in order to quantify the concentration of active ingredient present in each of the samples.

## Instrument Parameters

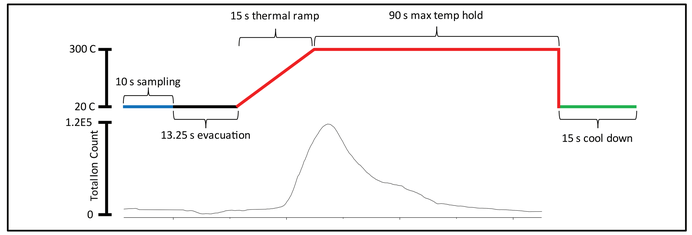


**Figure 1.** Thermal Desorption Method and observed TIC Desorption Profile for Lemon E-cigarette sample.

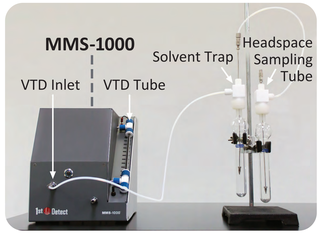


**Figure 2.** MMS-1000 in Headspace Sampling configuration.

**MS Parameters**
- Mass Range: 45 - 450 amu
- Scan Rate: 10Hz

**VTD Parameters - (Carbopack X sorbent)**
- Sampling Time: 10 s
- Desorption Time: 105 s
- Desorption Temperature: 300°C

## Results

### Quantification of Flavorants via Standard Addition

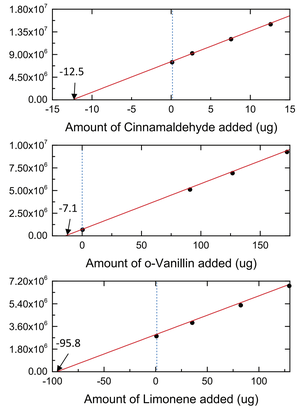


**Figure 3.** Standard Addition Calibration Curves for Cinnamaldehyde, o-Vanillin, and Limonene.

### Mass Spectrum and EIC of o-Vanillin

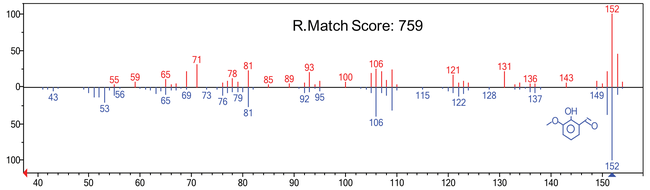


**Figure 4.** Mass Spectrum of o-Vanillin in E-Cigarette Liquid (Red) and o-Vanillin mass spectrum from NIST (blue). [1]

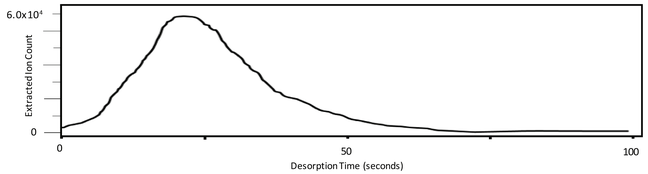


**Figure 5.** Extracted Ion Chromatogram (m/z 152) of o-Vanillin in E-Cigarette Liquid.

## Results (Cont.)

The table below shows the concentration of various flavorants found in the each of the samples. The concentration was derived by accounting for the sample size and the initial dilution of the neat E-Cigarette liquids. The characteristic spectral ion utilized for each flavorant was the molecular ion peak for that species.

| Chemical | Characteristic MS Peak (amu) | Mass of Analyte | Sample Size | Dilution Factor | Concentration | $R^2$ Fit |
|---|---|---|---|---|---|---|
| Cinnamaldehyde | 132.0 | 12.5 mg | 0.5 mL | 2.07 | 51.8 ppm | 0.973 |
| o-Vanillin | 152.0 | 7.1 mg | 0.5 mL | 2.07 | 29.4 ppm | 0.993 |
| Limonene | 135.0 | 95.8 mg | 0.5 mL | 2.07 | 396.6 ppm | 0.993 |

## Conclusions

This poster presents a novel method for quantifying various flavorings in E-Cigarette liquids using VTD-MS. The quick and easy headspace sampling method and standard addition approach provided excellent linearity and sensitivity for the target analytes. Results showed that levels of these flavorings were in the in the tens of micrograms per mL of E-Liquids. This approach demonstrated simplified sample preparation via aqueous dilution as well as rapid analysis (< 2 minutes) using headspace sampling.

## References

[1] NIST 14 Mass Spec Library